Increasing Charpy Impact Value of Polycarbonate (PC) Sheets Irradiated by Electron Beam

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Applying low-potential electron beam irradiation (EBI) dose of 0.13 MGy to polycarbonate (PC) sheet on both side surfaces apparently improved the impact value (a_{uc}) over the untreated samples at all accumulative probabilities of fracture (P_f). Based on 3-parameter Weibull equation, the 0.13 MGy-EBI also raised the lowest limit of a_{uc} (25.5 kJ m^{-2}) estimated at $P_f = 0$ (a_s) 14% over the untreated samples (22.0 kJ m^{-2}), indicating improved reliability and safety. EBI generates dangling bonds where repulsive forces are generated between the outer shell electrons at terminated atoms in the PC polymer irradiated near the sample surface, probably inducing relaxation of heterogeneous stress concentration, as well as compressive stress (high molecular density) by micro-expansion around terminated atoms increasing the a_{uc} . Increasing EBI dose increased ductility by decreasing the fractured split ratio, R_s , a characterization of separated portion of total cross-section of fractured PC. The 0.13 MGy dose ($R_s = 0.30$) appears to be at or near the optimal EBI dose obtaining the highest a_{uc} . Above 0.22 MGy the a_{uc} reduces with increasing dose. Excessive dangling bonds generation appeared to increase ductility in the form of lowering the R_s but weakens the molecular structure allowing chains to slide past each other easier decreasing impact values with less crack propagation. Therefore, when applying EBI to polycarbonate parts in industrial applications carefulness is highly recommended to adjust dose to optimal level for maximum strength and safety. [doi:10.2320/matertrans.M2018095]

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1. Introduction

The strengthening of light structural materials has been always required to develop high-speed transport vehicles with safety enhancement and low energy consumption. The structural formula of polycarbonate (PC) polymers includes two carbocyclic six-membered aromatic rings¹⁾ which are the main parts composing its strong molecular segments. PC exhibits high wear resistance as well as high strength and fracture toughness. It can be utilized for optical lenses, CD substrates, in electromagnetic wave shielding materials, as well as artificial body parts. When used as windows for vehicles, PC provides weight savings having lower density than glass. PC is also a promising material for electric car bodies reducing weight significantly improving efficiency eco-friendly to our environment. Therefore, further strengthening with reliability enhancement is always desired.

When the magnetic lens focuses the electron beam irradiation (EBI) with high voltage, it easily induces radiation damage in polymers as well as metals. On the other hand, homogeneously applying a lower dose on the order of 100 kV, referred to as homogeneous low-voltage electron beam (EBI),²⁻⁵⁾ makes it easy to migrate the surface atoms generating point defects in crystal and free volume in glassy materials. EBI has a positive record of improving many solid materials including mist resistance with wetting,²⁻⁵⁾ adhesion,⁶⁻¹⁵⁾ sterilization¹⁶⁾ and strengthening,^{1,17-30)} as well as both wear resistance and industrially utilized burnishing. Since the stability and homogeneity of industrial EBI equipment are tremendously high, they have been practically utilized for wallpaper sheet, building insulation and automotive parts up to several square meters in size.

In 2003, enhancements of bending fracture strength and its strain of epoxy polymer, generally utilized for matrix of carbon fiber reinforced polymer (CFRP) have been reported.²⁰⁾ Furthermore, for polycarbonate (PC) extremely low optimum dose of 0.02 MGy-EBI was found to enhance stiffness over 100%, upper yield stress $\sim 12\%$, lower yield stress $\sim 12\%$, tensile fracture strength $\sim 20\%$ and its strain $\sim 16\%$, although increased dose of more than 0.10 MGy-EBI decayed these mechanical properties.³⁰⁾

It follows high impact resistance is important to protect aircraft, automobiles and other applications from projectiles such as bird strike, volcanic rock or hailstone. Given the improvements in tensile properties of PC by EBI, it is hypothesized EBI can improve the impact resistance of the PC polymer 10 to 20% for practical use. Therefore, the purpose of the present work is to evaluate the effect of EBI on the impact value of the PC for application to aircraft windows and high-speed mover machines.

2. Experimental Procedure

2.1 Condition of EB-irradiation

Prior to HLEBI treatment, non-reinforced PC (TAKIRON INC, Tokyo) polymer samples were cut to dimensions $1.0 \times 80 \times 10 \text{ mm}$ (volume = 800 mm^3). The sheet samples were then homogeneously treated by the prototype equipment known as the electron-curtain processor (Type CB175/15/180L, Energy Science Inc., Woburn, MA), which was constructed with a vacuum chamber (240 mm in diameter under 10^{-5} Pa) with a tungsten filament (215 mm length) and a thin titanium window (150 mm length, 150 mm width and $0.01 \mu\text{m}$ thickness), and sample treatment compartment with conveyer under 1 atm N_2 (g).^{1-15,17-30)}

The sheet specimen was homogeneously irradiated with the electron beam through the titanium thin film window attached to the vacuum chamber. The tungsten filament in vacuum was used to generate the electron beam at a low energy (acceleration potential, V : kV), of 170 kV and irradiating current density (J : Am^{-2}) of 0.089 Am^{-2} . Although the electron beam generation was in a vacuum, the irradiated sample was kept under protective 1 atm N_2 (g). The distance between sample and window was 35 mm. To prevent oxidation, the samples were kept in the protective 1 atm N_2 with an O_2 residual concentration below 400 ppm. The flow rate of the N_2 was 1.5 L s^{-1} at 0.1 MPa of N_2 gas pressure.

Each sweep of irradiation dose (0.043 MGy (kJ g^{-1})) was applied for only a short time (0.23 s) to avoid excessive heating of the sample; the temperature of the sample surface remained below 323 K just after irradiation. The sample in the aluminum plate holder ($0.15 \text{ m} \times 0.15 \text{ m}$) was transported on a conveyor at a speed of 10.0 m min^{-1} . The sheet electron beam irradiation was applied intermittently. Repeated irradiations to both side surfaces of the samples were used to increase the total dose of irradiation. The interval between the end of one period of irradiation sweep and the start of the next was 30 s .

When the irradiation current (I : mA), the conveyor speed (S : m min^{-1}), and number of irradiations (N) are determined, the irradiated dose (D : MGy) is expressed by the following equation:

$$\text{Dosage (MGy)} = 0.216 \times (I/S) \times N \quad (1)$$

The irradiation dose has been controlled by the integrated irradiation time in each of the samples. Here, irradiation dose has been corrected by using an FWT nylon dosimeter of RCD radiometer film (FWT-60-00: Far West Technology, Inc. 330-D South Kellogg Goleta, California 93117, USA) with an irradiation reader (FWT-92D: Far West Technology, Inc. 330-D South Kellogg Goleta, California 93117, USA). The dose is 0.0432 MGy at each irradiation. Based on the mean density (ρ : kg m^{-3}) and irradiation potential at the specimen surface (V : keV), the penetration depth (D_{th} : μm) of EB-irradiation is expressed by the following equation.²⁰⁾

$$D_{\text{th}} = 66.7V^{5/3}/\rho \quad (2)$$

Specimen surface electrical potential (V) was mainly reduced going through the Ti window (ΔV_{Ti}) and N_2 gas atmosphere (ΔV_{N_2}).

$$V = 170 \text{ keV} - \Delta V_{\text{Ti}} - \Delta V_{\text{N}_2} \quad (3)$$

Based on eq. (2), the dropped potential values, ΔV_{Ti} and ΔV_{N_2} are estimated from the acceleration potential (170 keV), the $13 \mu\text{m}$ thickness (T_{Ti}) of the titanium window (density: 4540 kg m^{-3}), and the 25 mm distance between the sample and the window (T_{N_2}) in the N_2 gas atmosphere (density: $\rho_{\text{N}_2} = 1.13 \text{ kg m}^{-3}$).

$$\begin{aligned} \Delta V_{\text{Ti}} &= T_{\text{Ti}}/D_{\text{thTi}} \times 170 \text{ keV} \\ &= T_{\text{Ti}}\rho_{\text{Ti}}/[66.7 \times (170 \text{ keV})^{2/3}] \\ &= (10 \mu\text{m}) \times (4540 \text{ kg m}^{-3})/[66.7 \times (170 \text{ keV})^{2/3}] \\ &= 22.2 \text{ keV} \end{aligned} \quad (4)$$

$$\begin{aligned} \Delta V_{\text{N}_2} &= (T_{\text{N}_2}/D_{\text{thN}_2}) \times V_{\text{Ti}} \\ &= T_{\text{N}_2}\rho_{\text{N}_2}/[66.7 \times (170 \text{ keV} - \Delta V_{\text{Ti}})^{2/3}] \\ &= (25 \times 10^3 \text{ m}) \\ &\quad \times (1.13 \text{ kg m}^{-3})/[66.7 \times (170 \text{ keV} - 22.2 \text{ keV})^{2/3}] \\ &= 15.2 \text{ keV} \end{aligned} \quad (5)$$

Since the dropped potential values are 22.2 keV and 15.2 keV , the specimen surface electrical potential, V is obtained to be 132.6 keV as follows.

$$V = 170 \text{ keV} - 22.2 \text{ keV} - 15.2 \text{ keV} = 132.6 \text{ keV} \quad (6)$$

Based on the eq. (2) and the density is 1.20 g cm^{-3} of the PC samples, the penetration depth (D_{th} : μm) is $191 \mu\text{m}$.

2.2 Samples and Charpy impact test

Usually, damage by a hemispherical impactor is initiated as a point force on the center of a square or rectangular specimen ~ 70 to 150 mm^2 by slow point force,³¹⁾ drop tower, or projectile.^{32–35)} For low energy tests, impact is followed by non-destructive testing (NDT) with an ultrasonic transducer to detect delamination undetectable to the eye which can reduce strength significantly. Compression after impact (CAI) testing is often conducted after point impact in aerospace materials such as quasi-isotropic CFRP for safety design,^{36,37)} and is generally utilized to evaluate safety against impact accidents such as bird strike, volcanic rock or hailstone on aircraft CFRP.^{38,39)} On the other hand, Charpy impact test employs a drop-weight pendulum and evaluates impact absorption characteristics and relative impact toughness of materials often used in quality control applications utilized as a low-cost and rapid way to estimate reaction to higher velocity impact. We do not claim the Charpy test to be a substitute for point impact followed by compression after impact (CAI). However, Charpy impact method could possibly be used as an inexpensive preliminary evaluation to screen candidate materials to later test with indentation or projectile followed by CAI. Hence, Charpy may give a rough or better estimation of which materials and what temperatures a projectile such as bird strike, volcanic rock or hailstone will cause the most damage. Tests are carried out calibrating for air friction and effect of air temperature and humidity on the swing and pivot of the pendulum. Therefore, the velocity, v (ms^{-1}) and kinetic energy, KE (J) hitting the sample are assumed to be approximately constant similar to comparing projectiles in a point impact test hitting the composite surface at constant v and KE . Thus, when the Charpy impact test impact velocity, v hitting the sample is calculated as:⁴⁰⁾

$$v = [2gR(1 - \cos\alpha)]^{0.5} \quad (7)$$

where g is gravitational constant (9.8 ms^{-2}), R is length (m) of hammer weight point from rolling center (0.21 m), and α is start angle before impact (2.3 Radians , 132°), then v of the hammer hitting the specimen is $\sim 1.74 \text{ ms}^{-1}$ ($\sim 3.89 \text{ mi/hr}$). Potential energy (PE) (J) of impact is:⁴⁰⁾

$$PE = R(1 - \cos\alpha)F \quad (8)$$

where F is measured supporting force (N) exerted by the pendulum in the horizontal position. PE is assumed to be equivalent to the KE in the horizontal vector, $0.5mv^2$, or 1.30 J ($1.3 \times 10^{-3} \text{ kJ}$). The surface area of the impactor tip

is small with circular shaped ~ 0.5 mm diameter, so KE/A is large.

In order to evaluate the impact fracture toughness, the Charpy impact values of the PC samples using a standard impact fracture energy measurement system (Shimadzu Corporation No. 51735) (JIS K 7077)⁴¹⁾ was utilized.^{1,21–30)} EBI were performed on both side surfaces of the PC samples.

The impact fracture energy (E) is expressed by eq. (9):²⁹⁾

$$E = WR[(\cos \beta - \cos \alpha) - (\cos \alpha' - \cos \alpha)(\alpha + \beta)/(\alpha + \alpha')] \quad (9)$$

Here, E , W , R , β , α and α' are impact fracture energy (J), hammer weight (8.43 N), length (m) of hammer weight point from rolling center, the maximum angle after impact, start angle before impact, and the maximum angle of the blank test (for calibration), respectively. The Charpy impact value (a_{uc} : kJ m^{-2}) is expressed by the following equation.²⁹⁾

$$a_{uc} = E/(bt) \times 10^3 \quad (10)$$

Here, E , b ($= 10 \pm 0.2$ mm) and t ($= 2.8$ mm) are impact fracture energy (J), sample width (mm), and span distance (sample thickness, mm), respectively. The distance between supporting points was 40 mm.

Evaluating the cumulative probability of fracture (P_f) is a convenient method of quantitatively analyzing experimental values, and in industry is often employed in statistical quality control (QC). P_f is expressed by the following equation which is a generalized form of the median-rank method:⁴²⁾

$$P_f = (i - 0.3)/(N_s + 0.4) \quad (11)$$

Here N_s and i are total number of samples and rank order integer of a_{uc} of each sample, respectively where i is from weakest to strongest. In this case, $N_s = 9$ hence when i values are 1, 5, and 9, their corresponding P_f values are 0.07, 0.50 and 0.93, respectively.

3. Results

Figure 1 shows that 0.13 MGy-EBI to the PC polymer apparently improves the impact value (a_{uc}) $\sim 21\%$, from 24.6 to 29.8 kJ m^{-2} over the untreated samples at median- P_f of 0.50; and from 22.0 to 26.0 kJ m^{-2} at low- P_f of 0.07 indicating reliability of parts is possible. At high- P_f of 0.93, a_{uc} is improved 11.3% from 44.0 to 49.0 kJ m^{-2} . Furthermore, the 0.13 MGy-EBI apparently improves the a_{uc} at all P_f , while exhibiting a 24% improvement in average a_{uc} from 27.3 to 33.8 kJ m^{-2} .

Although Fig. 1 shows a_{uc} is reduced at doses above 0.22 MGy, the 0.13 MGy-EBI appears to be near the optimal dose for the PC as shown in Fig. 1 since the lower and higher EBI doses of 0.04 and 0.22 MGy resulted in unchanged a_{uc} at low P_f of 0.07 and decreases at the high- P_f of 0.93; while higher EBI doses decrease the a_{uc} further.

4. Discussion

4.1 The lowest impact value, a_s , estimated

The a_s , a_{uc} value at $P_f = 0$ represents the statistically lowest impact value for safety considerations and deems valuable for process with quality control. By applying the

Fig. 1 Changes in experimental impact values, a_{uc} (kJ m^{-2}) of PC at each dose of EBI against P_f , together with the lowest impact values calculated ($a_s = a_{uc}$ at $P_f = 0$).

0.13 MGy EBI to the PC, Fig. 1 shows the a_s estimated by 3-parameter Weibull equation is improved 14% over the untreated from 22.0 to 25.5 kJ m^{-2} .

Since the experimental a_{uc} values at lower P_f value have largely deviated from the linear relationship estimated by 2-parameter Weibull equation,^{26,43)} the practical a_{uc} value at extremely low P_f near zero cannot be precisely estimated.^{26,43)}

Hence, when the statistical 3-parameter Weibull equation is assumed to be applicable to the measured a_{uc} value, the P_f value depends on the risk of rupture ($[a_{uc} - a_s]/a_{III}$), as a following equation.^{26,43)}

$$P_f = 1 - \exp[-([a_{uc} - a_s]/a_{III})^m] \quad (12)$$

The linear logarithmic form can then be obtained:

$$\ln[-\ln(1 - P_f)] = m \ln[a_{uc} - a_s] - m \ln a_{III} \quad (13)$$

In predicting the required impact value of materials, the a_{uc} value at $P_f = 0$ (a_s), the constant (a_{III}) and 3-parameter Weibull coefficient (m) are key parameters. When the term ($\ln[-\ln(1 - P_f)]$) is zero, the a_{III} is the a_{uc} .

Figure 2 shows iteration of the linear logarithmic form in eq. (13) for the potential lowest impact value (a_s) to get the maximum correlation coefficient (F) corresponding to the a_s of 22.0, 22.0, 25.5, 9.0, and zero kJ m^{-2} (arrows) for the untreated, 0.04, 0.13, 0.22 and 0.30 MGy-EBI irradiated samples, respectively, showing high linear correlations of 0.987, 0.972, 0.977, 0.977 and 0.956. The resulting linear relationships are plotted in Fig. 3. Note, the a_s at $P_f = 0$ are also indicated in Fig. 1 showing the highest was obtained from the 0.13 MGy-EBI samples. Figures 1 and 2 show at 0.22 MGy and above, the a_s at $P_f = 0$ was reduced. Nevertheless, our results show the 0.13 MGy-EBI can improve the a_{uc} at $P_f = 0$ (a_s).

Figure 4 shows the 0.13 MGy-EBI was able to successfully enhance the PC polymer across the fracture probability range for selected P_f , respectively along with the a_s at $P_f = 0$. For doses above 0.13 MGy drops in a_{uc} with increasing EBI dose indicate the safety consideration needed when optimizing EBI dose.

Fig. 2 Changes in the F value of PC at each dose of EBI against the potential lowest impact value, e_{a_s} .

Fig. 3 Relationships between $\ln(a_{uc} - a_s)$ vs. $\ln[-\ln(1 - P_f)]$ for PC at each dose of EBI.

4.2 Optical observations and fractured split ratio, R_s , of fractured PC

Observation of the PC samples showed they were partially separated by the impact, exhibiting simple bending fracture at the compressive side accompanied by cracks of a certain area fraction propagating into the thickness from the tensile side.

Therefore, to assess effects of EBI level on brittleness or ductility of the PC samples, we define the fractured split ratio (R_s) here as separated part of possible total impacted cross-section:

$$R_s = (S_0 - S)/S_0 \quad (14)$$

where S_0 and S are total cross-sectional area and non-deformed (undamaged) area, respectively. The lower the R_s , the more ductile the fracture mode. Measured split ratio R_s , for the 0.13 MGy-EBI samples is significantly lower (0.30 ± 0.10) than that of the untreated ($\sim 0.70 \pm 0.10$) but higher than that (0.15 ± 0.05) of the 0.86 MGy samples. Thus, increasing EBI dose from zero to 0.86 MGy improves the ductility. However, the 0.13 MGy-EBI appears to produce

Fig. 4 Changes in Charpy impact values, a_{uc} (kJm^{-2}) of PC at each selected P_f , of 0.07, 0.50 and 0.93 against dose (MGy) of EBI.

optimal ductility as evidenced by yielding the highest impact values, a_{uc} hence being able to withstand maximum absorbed impact energy. At higher doses of 0.86 MGy, a_{uc} is lowered.

Figures 5(a) and (b) show photographs of fractured PC samples untreated and treated at 0.13 MGy-EBI at median accumulative probability ($P_f = 0.50$). Figure 5 shows when impacted (arrows) in the 0.13 MGy sample (b) the crack penetrates at lower depth into the thickness from the tensile side than that of the untreated (a). Crack origins at the tensile side of all specimens are observed to occur accompanied by white crazing. At the compressive side in the untreated and 0.13 MGy samples the white crazing at the impact point was observed to occur, while in the 0.86 MGy samples simple ductile fracture occurs.

4.3 Effects of EBI on dangling bonds and impact values of PC

Dangling bonds have been reported to be detected in PC by 0.0216 MGy-EBI irradiation dose as a sharp ESR (electron spin resonance) peak.³⁰⁾ In contrast, it is reported untreated PC does not produce an ESR peak, only a flat line hence no dangling bonds.³⁰⁾ ESR is applied to detect unpaired electrons by their spins ($m_s = \pm 1/2$). Repulsive forces are generated between the outer shell electrons at terminated atoms irradiated near the sample surface inducing relaxation of heterogeneous stress concentration, as well as compressive stress by micro-expansion around terminated atoms increasing the a_{uc} .³⁰⁾ However, the high EBI dose of 0.86 MGy while increasing ductility apparently generates excessive dangling bonds allowing chains to slide past each other easier weakening the molecular structure decreasing resistance to impact with less crack propagation. Therefore, the optimum 0.13 MGy-EBI dose generates the optimum dangling bond density to create micro-compressive forces between atoms increasing impact resistance.

Figure 6 shows schematic illustrations of PC before and after EBI showing terminated atoms with dangling bonds generating repulsive forces³⁰⁾ at high molecular density zone of the polymers. These repulsive forces generate micro-expansive stress around dangling bonds sites within and between the PC polymer chains strengthening the PC matrix.

Fig. 5 Photograph of side surfaces of impacted PC samples showing that of (a) untreated and; (b) 0.13 MGy-EBI treated, both at median accumulative probability ($P_f = 0.50$). Arrows indicate impact direction.

As shown in Figs. 1 and 4, the 0.13 MGy-EBI dose appears to generate the optimum dangling bond density for increasing the a_{uc} .

Since dangling bond densities in PC are controlled by EBI dose, it is possible to get optimal density of dangling bonds to enhance the a_{uc} . Obtaining the optimum dose of EBI appears to be a useful tool for quick strengthening to get high impact value of PC. However, carefulness is needed in quality control to adjust EBI dose to optimal level for each given situation when treating aircraft and other parts to maximize strength with high safety.

5. Conclusions

- (1) Applying electron beam irradiation (EBI) dose of 0.13 MGy to PC polymer samples on both side surfaces homogeneously improved the Charpy impact value (a_{uc}) 21%, from 24.6 to 29.8 kJ m⁻² over the untreated samples at median- P_f of 0.50; and at low- P_f of 0.07 improved 18% from 22.0 to 26.0 kJ m⁻² indicating

Fig. 6 Schematic nanoscale illustration of polycarbonate polymer before and after EBI.

reliability of parts is possible. At high- P_f of 0.93, a_{uc} was improved 11.3% from 44.0 to 49.0 kJ m⁻². The 0.13 MGy-EBI also apparently improved the a_{uc} at all P_f over the untreated samples while exhibiting a 24% improvement in average a_{uc} from 27.3 to 33.8 kJ m⁻².

- (2) For safety, reliability and quality control considerations the 0.13 MGy-EBI raised the lowest limit of impact value (a_s) at $P_f = 0$ estimated by the 3-parameter Weibull equation 14% over the untreated samples from 22.0 to 25.5 kJ m⁻².
- (3) Hence, the 0.13 MGy-EBI appears to be near the optimal dose for Charpy impact resistance of the PC samples raising a_{uc} values above those of 0.04 and 0.22 MGy, and 0.30 to 0.86 MGy.
- (4) EBI generates dangling bonds where repulsive forces are generated between the active shell electrons at terminated atoms in the PC polymers irradiated near the sample surface, probably inducing relaxation of heterogeneous stress concentration, as well as compressive stress (high molecular density) by micro-expansion around terminated atoms increasing the a_{uc} .
- (5) Optical observation of PC sample fracture surfaces showed increasing EBI dose increased ductility by decreasing the fractured split ratio, R_s a characterization of the separated portion of the total cross-section from untreated ($R_s = 0.70$), to 0.86 MGy ($R_s = 0.15$) as cracks propagates into the thickness from the tensile side. Since in the literature EBI is reported to generate dangling bonds in PC creating micro-expansive forces increasing mechanical properties, the 0.13 MGy dose gave the optimum dangling bond density for impact strength. However, at higher EBI doses above 0.22 MGy, excessive dangling bond generation appeared to increase ductility in the form of lowering the split ratio, R_s but weakening the molecular structure allowing chains to slide past each other easier decreasing impact values with less crack propagation. Therefore, carefulness is extremely necessary in quality control to adjust the EBI dose to optimal level when treating for PC aircraft and other parts to maximize safety with high strength.

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