Effects of Homogeneous Irradiation of Electron Beam with Low Potential on Microscopic Fracture Resistance of Borosilicate Glass Surface

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An effect of homogeneous irradiation of electron beam with low potential (HIEBL) on the micro-fracture resistance of borosilicate glass was studied by using indentation of hardness test. 0.04 MGy-HIEBL increased the critical resistant energy (E_f) of micro plastic deformation to generate the crack and tremendously decreased their experimental errors. Furthermore, the high values of the midpoints (E_f^{mid}) of E_f values were obtained from 0.04 to 0.22 MGy of irradiation dose. If HIEBL annihilated the spontaneous crack origins, the minimum E_f value (E_f^{min}) before irradiation was the critical resistant energy of micro-plastic deformation to propagate the crack (E_f^{p}). Since HIEBL of less than 0.43 MGy didn't affect the fracture toughness (K_{IC}) estimated by fracture toughness to propagate the crack using standard indentation fracture method, the E_f^{p} value of the borosilicate glass didn't depend on the irradiation dose. On the other hand, the critical resistant energy of micro-plastic deformation to generate the crack (E_f^{n} ; $E_f^{\text{n}} = E_f - E_f^{\text{p}}$) was defined. Thus, HIEBL increased the E_f values because of increasing E_f^{n} values. Based on the increasing the height of electron spin resonance (ESR) spectra, HIEBL formed dangling bonds in silica glassy network structure, as well as reformed the crack tip from sharp to dull. Thus, they probably relaxed the residual strain. If the partial relaxation of the residual strain mainly occurred around these dangling bonds of the silicon-oxygen pairs in the network structure, the increased E_f value was mainly due to the relaxation of the network structure, as well as the annihilation of crack origin. Therefore, HIEBL prevented the crack generation.

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1. Introduction

Strengthening of transparent thin sheet glass is an important technique in the manufacture of large-area liquid-crystal displays of televisions.¹⁾ However, it is easy to break the thin broad glass.²⁾ The borosilicate glass can be used as an electronic insulator³⁾ and biological knife. However, the weak fracture toughness of borosilicate glass is also a serious problem when it is used in the form of sheets or fine needles.

Although the ductile fracture is generally found in metals, the brittle fracture, which is composed of nucleation and propagation of cracks,^{4,5)} is observed in steels with body centered cubic structure and ceramics by deformation with lower temperature and high strain rate. Since it is perfectly difficult to annihilate the crack origins, strengthening is often controlled by crack propagation.⁵⁻⁷⁾ General strengthening has been performed by fiber reinforcement⁵⁾ and transformation induced toughness.⁶⁾ Furthermore, it is a useful tool to add residual compressive strain by chemical reinforcement process.⁷⁾ However, process conditions, related to pressure, composition, temperature and time which are dependent on the materials, must be complexly selected.

On the other hand, the shot-peening has been a useful tool to randomize the metallic glasses,⁸⁾ enhances their saturated magnetization⁹⁾ and softens them.¹⁰⁾ However, in ceramics it is difficult that the shot-peening to randomizes the surface ceramics without fractures.

In order to randomize the glassy structure without fracture, the irradiation of electron beam, which is the limited small mass, has been used as the ball of shot-peening.¹¹⁻¹³⁾ Homogeneous irradiation of electron beam with low potential (HIEBL) generates dangling bonds,^{14,15)} as well as annihi-

lates the crack, resulting in the strengthening.^{3,11-13)} The high stress resistance to propagate cracks and to nucleate crack origins macroscopically dominates the high fracture resistance. However, influence of HIEBL on the fracture resistance has not been reported.

Therefore, effects of HIEBL on the micro-fracture resistance (elasticity, fracture toughness, the critical resistant energy (E_f) of micro plastic deformation to generate the crack) of borosilicate glass were studied by using indentation of hardness test.

In addition, the critical resistant energy of micro-plastic deformation to propagate the crack (E_f^{p}) and the critical resistant energy of micro-plastic deformation to nucleate the crack (E_f^{n}) were obtained.

2. Experimental Procedure

2.1 Homogeneous irradiation of electron beam

The borosilicate glass sheets (Kobayashi Tokushu Glass, Co. Ltd. Japan) were homogeneously irradiated by using an electron-curtain processor (Type CB175/15/180L, Energy Science Inc., Woburn, MA, Iwasaki Electric Group Co. Ltd. Tokyo).^{3,11-13)} The specimen was homogeneously irradiated with an electron beam through a titanium window attached to a 240-mm-diameter vacuum chamber. A tungsten filament in a vacuum was used to generate the electron beam with an electrical potential of 170 kV and an irradiating current of 2.0 mA. To prevent their oxidation, the samples were kept under a nitrogen atmosphere of 0.1 MPa with a residual concentration of oxygen of less than 400 ppm. The flow rate of the nitrogen gas was 1.5 L/s.

Christenhusz and Reimer¹⁶⁾ evaluated the depth of penetration of EB irradiation of less than 200 kV electrical potential by measuring the Joule heat generated in copper films.¹⁶⁾ Based on the density (ρ : kg/m³) and electrical

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potential (V : kV), the penetration depth (D_{th} : m) was given by the following equation:

$$D_{th} = 66.7 V^{5/3} / \rho \quad (1)$$

The electrical potential (128 keV) was estimated from the electrical potential (170 keV), the thickness [1.0×10^{-5} m (10 μ m)] of the titanium window [$\rho = 4540$ kg/m³ (4.54 g/cm³)], and the distance (30 mm) in the nitrogen gas atmosphere ($\rho = 1.13$ kg/m³) between the surface of the sample and the window. On the basis that the electrical potential is 128 keV, eq. (1) predicts that the penetration depth (D_{th} : m) should be 99.3 μ m in the case of the borosilicate sample ($\rho = 2230$ kg/m³).

On the other hand, the penetration depth of EB irradiation related to the mass thickness (l_0 : mg/cm²) and irradiation voltage (E : kV), is expressed by the following equation suggested by Libby.¹⁷⁾

$$l_0 = 1/150 \times E^{5/3} \quad (2)$$

The estimated mass thickness is 34.8 mg/cm², when initial irradiation voltage was 170 keV. Since the mass thickness of Ti foil (1.78 mg/cm²) and N₂ gas (0.15 mg/cm²) reduce the penetration depth of EB irradiation, the mass thickness of borosilicate glass sample was 32.9 mg/cm². In addition, the electrical potential on sample surface was expressed by the following equation.

$$E = (150 \times l_0)^{3/5} \quad (3)$$

Furthermore, the penetration depth of the glass was obtained about 145.74 μ m by assumptions of Libby.¹⁷⁾

The sample, positioned in an aluminum plate holder (0.15 $\times$ 0.15 m), was transported on a conveyor at a speed of 9.56 m/min. Because the minimum dose of EB irradiation was 43.2 J/g ($= 1.13 \times 10^{13}$ em⁻² = 0.0432 MGy), each burst of irradiation was performed for a short time (0.23 s) at 0.0432 MGy to avoid excessive heating of the sample: the temperature of the sample surface remained below 323 K just after irradiation. Both surfaces of the samples were repeatedly irradiated to increase the total irradiation dose. The interval between the end of one period of irradiation and the start of the next was 30 s. The dosage was proportional to the yield value determined from the irradiation current, the conveyor speed, and number of irradiations. The yield value was calibrated by means of FWT nylon dosimeters (Far West Technology, Inc., Goleta, CA, USA).

The electron number density (em⁻²) was expressed by the following equation in metals.¹⁸⁾

$$Ng = DE/Cg \quad (4)$$

where, Ng , DE and Cg were the irradiation dose (em⁻²), the dose equivalent (Sv/s) and Transferred coefficient of particle flux-dose equivalent [0.1649×10^{-13} (Sv/s)/(em⁻²)],¹⁸⁾ respectively.

2.2 Measurements of elasticity and hardness

Elasticity of ceramics was generally measured by bending test. However, it was difficult to measure the elasticity of surface layer of irradiated samples. To obtain the elasticity at sample surface, the elasticity was expressed by the following equation.¹⁹⁾

$$HK/E = 0.312 - 2.22(b'/a') \quad (5)$$

where, HK , E , b' and a' were, the Knoop hardness (GPa), the elasticity (GPa), the length of minor axis (m) and the length of major axis (m), respectively.

In addition, the Knoop (load (P) = 1.96 N) and Vickers' (0.49 N < P < 2.94 N) hardness values were evaluated by using standard hardness measurement systems of ASTM C1326-99, ASTM C1327-99 and indentations without crack at corner.

2.3 Measurements of fracture toughness

To evaluate the fracture toughness, the fracture toughness of the borosilicate glass samples with and without EB irradiation was measured by using a standard indentation micro fracture measurement.²⁰⁾ The fracture toughness was expressed by the following equation.²⁰⁾

$$K_{IC} = 0.026 E^{1/2} P^{1/2} a / C_L^{3/2} \quad (6)$$

where, K_{IC} , E , P , a , and C_L were the fracture toughness (MNm^{-3/2}), the elasticity (Pa), the load (N), the half value of diagonal distance (m) and the half value of crack length (m), respectively.

2.4 Measurements of critical load (P_f) and diagonal (d_f) to generate cracks

To evaluate critical resistant energy (E_f) on fracture, the experimental value of the critical indentation diameter (d_f : fracture strain), and critical fracture load (P_f : fracture stress) were obtained, using a micro-Vickers indentation tester.^{11,21)} The relationship between load (P) and diagonal distance (d) of indentation was obtained. The maximum values ($P_f^{\max}$ and $d_f^{\max}$) of P and d were determined from indentations that were without cracks. On the other hand, the minimum values P and d of ($P_f^{\min}$ and $d_f^{\min}$) were determined from indentations that were observed with cracks. The midpoints of critical indentation diameter (d_f^{mid}) and critical load (P_f^{mid}) on fracture were defined and expressed by following equations.^{11,21)}

$$d_f^{\text{mid}} = (d_f^{\min} + d_f^{\max})/2 \quad (7)$$

$$P_f^{\text{mid}} = (P_f^{\min} + P_f^{\max})/2 \quad (8)$$

2.5 Electron spin resonance (ESR) measurement

To obtain more-precise information on atomic-scale structural changes in the glass, the density of the dangling bonds was measured by means of an electron spin resonance (ESR) spectrometer (JES-FA2000, Nippon Denshi Ltd., Tokyo).^{11,22)} The microwave frequency range used in the ESR analysis was the X-band at 9.45 ± 0.05 GHz with a field modulation of 100 kHz. The microwave power was 1 mW. The magnetic field was varied from 318.7 to 328.7 mT. The spin density was calculated by using a Mn²⁺ standard sample. Only ESR spectra, instead of spin densities, are given. Based on the standard calibration material [4-hydroxy-2,2,6,6-tetramethyl piperidine-1-oxyl (TEMPO, 089-04191, Wako Pure Chemical Industries Ltd., Tokyo)] and Mn²⁺ in the MnO the density of dangling bond was estimated by double integration of intensity of ESR signal.²²⁾

Fig. 1 Changes in Knoop ($\triangle$: $P = 1.96$ N) and Vickers hardness ($\circ$: $P = 1.96$ N, $\diamond$: mean value (0.49 N $< P < 2.94$ N)) of borosilicate glass against irradiation dose of HIEBL.

Fig. 2 Changes in elasticity of borosilicate glass against EB irradiation dose.

3. Results

3.1 Influence of HIEBL on hardness and elasticity

Figure 1 shows changes in the Knoop and Vickers' hardness values ($P = 1.96$ N) against irradiation dose. Although mean values (0.49 N $< P < 2.94$ N) of Vickers' hardness value gradually increase with increasing irradiation dose, HIEBL doesn't affect the hardness values at 1.96 N load. The bold solid line means approximate linear relationship through several mean values of Vickers hardness ($\diamond$).

Figure 2 shows irradiation dose dependence of the elasticity (E) estimated by eq. (5). HIEBL of less than 0.5 MGy doesn't change the elasticity, as well as the values ($P = 1.96$ N) of Knoop hardness.

3.2 Influence of HIEBL on fracture toughness (K_{IC})

Figure 3 shows change in the fracture toughness (K_{IC}), which is estimated by fracture toughness to propagate the crack using standard indentation fracture (see eq. (6)),

Fig. 3 Changes in fracture toughness of borosilicate glass against EB irradiation dose.

Fig. 4 Changes in load, P against diagonal, d of borosilicate glass before and after 0.22 MGy-EB irradiations.

against dose of HIEBL. The broken line means K_{IC} value before HIEBL. HIEBL of less than 0.5 MGy is confirmed not to decrease the fracture toughness (K_{IC}).

3.3 Influence of HIEBL on the critical resistant load (P_f) of micro plastic deformation to generate the crack

Relationship between diagonal ($d/\mu\text{m}$) and load (P/N) is expressed by a following equation.²³⁾

$$\log P = \log P_0 + n \log d \quad (9)$$

Here, P_0 and n are the Meyer's index and constant, respectively.

Figure 4 shows the relationship between diagonal ($d/\mu\text{m}$) and load (P/N) of indentation of borosilicate glass before and after irradiation. Based on the least-squares method, a solid line (0.49 N $< P < 2.94$ N) is drawn by using experimental values of indentations without cracks. Although the experimental values of indentations with cracks often deviate the linear relationship, the linear relationships of experimental values of indentations without crack exhibit the high correlation coefficient.

Fig. 5 Changes in the maximum ($P_f^{\max}$), mid point (P_f^{mid}) and minimum ($P_f^{\min}$) values of critical resistant load (P_f) of micro plastic deformation to generate the crack of borosilicate glasses against irradiation dose.

Although the lowest critical load ($P_f = 0.74$ N) generates cracks (●) occurred at corners of the indentation ($d_f = 13.5 \mu\text{m}$) in the borosilicate glass before irradiation, 0.22 MGy-HIEBL enhances the lowest critical load ($P_f = 1.47$ N) to generate the crack at corners of the indentation (▲: $d_f = 18.7 \mu\text{m}$). Namely, 0.22 MGy-HIEBL improves the resistance to crack generation.

Figure 5 shows changes in the maximum ($P_f^{\max}$), midpoint (P_f^{mid}) and minimum ($P_f^{\min}$) values of critical resistant load (P_f) of micro plastic deformation to generate the crack against irradiation dose. Although 0.04 MGy-HIEBL slightly decreases the $P_f^{\max}$ value, it increases the P_f^{mid} and $P_f^{\min}$ values. Since 0.04 MGy-HIEBL tremendously decreases the experimental errors of P_f values, it shows that 0.04 MGy-HIEBL homogenizes the borosilicate glass.

The maximum values of $P_f^{\max}$ and $P_f^{\min}$ values are found at 0.22 and 0.04 MGy of irradiation dose, respectively. The high constant values of P_f^{mid} are obtained from 0.04 to 0.22 MGy of irradiation dose.

On the other hand, HIEBL from 0.22 to 0.43 MGy decreases the $P_f^{\max}$ and P_f^{mid} values. HIEBL from 0.04 to 0.43 MGy also decreases the $P_f^{\min}$ values, although they are more than that before irradiation.

3.4 Influence of HIEBL on the critical resistant diagonal (d_f) of micro plastic deformation to generate the crack

Figure 6 shows changes in the maximum ($d_f^{\max}$), midpoint (d_f^{mid}) and minimum ($d_f^{\min}$) values of critical resistant diagonal of micro plastic deformation (d_f) to generate the crack against irradiation dose. Although HIEBL at 0.04 MGy slightly decreases the $d_f^{\max}$ value, it increases the d_f^{mid} and $d_f^{\min}$ values. Since 0.04 MGy-HIEBL tremendously decreases the experimental errors of d_f values, it shows that HIEBL at 0.04 MGy homogenizes the borosilicate glass.

The maximum values of $d_f^{\max}$ and $d_f^{\min}$ values are found at 0.22 and 0.04 MGy of irradiation dose, respectively. The high values of d_f^{mid} are observed from 0.04 to 0.22 MGy of irradiation dose.

Fig. 6 Changes in the maximum ($d_f^{\max}$), midpoint (d_f^{mid}) and minimum ($d_f^{\min}$) values of critical resistant diagonal (d_f) of micro plastic deformation to generate the crack of borosilicate glasses against irradiation dose.

On the other hand, HIEBL from 0.22 to 0.43 MGy apparently decreases the $d_f^{\max}$ and d_f^{mid} values. HIEBL from 0.04 to 0.43 MGy also decreases the $d_f^{\min}$ values, although they are more than that before irradiation.

3.5 Dangling bonds formation by EB-irradiation

Figure 7 shows the ESR signals of silica and borosilicate glasses before and after EB irradiation. Although no ESR signals can be detected from untreated sample, a sharp ESR signal near the broken line in Fig. 7 is observed in the irradiated silica glass, corresponding to dangling bonds of an E-prime center consisting of a Si-O pair.^{14,15)} In silica glass, each silicon atom is generally coordinated to four atoms of oxygen, whereas a silicon atom with a dangling bond is coordinated to three atoms of oxygen: A silicon atom site with a dangling bond in silica glass is defined as an E-prime center. HIEBL at 0.22 MGy enhances the intensity of the ESR signal, showing that dangling bonds are formed in the silica glass.²²⁾

On the other hand, the irradiated borosilicate glass shows many ESR signals at different magnetic field values of the top peak, as shown in Fig. 7. Furthermore, additional irradiation at 0.43 MGy enhances the intensity of the ESR signal, which is higher than that at 0.22 MGy. Thus, the additional irradiation enhances the density of dangling bonds in borosilicate glass samples.

4. Discussion

4.1 Critical resistant energy (E_f) of micro plastic deformation to generate the crack

To evaluate the effects of EB irradiation on fracture toughness in borosilicate glass, the critical resistant energy (E_f) on fracture is obtained by using experimental values of critical indentation diameter (d_f) and critical fracture load (P_f).^{11,21)}

Fig. 7 ESR signals of silica and borosilicate glasses before and after EB irradiation.

$$E_f = C \times P_f \times d_f/3 \quad (10)$$

Here, C is a constant ($C = h$ [indentation depth]/ d [indentation diameter] = 0.143).

Based on the $P_f^{\max}$, P_f^{mid} , $P_f^{\min}$, $d_f^{\max}$, d_f^{mid} and $d_f^{\min}$ values and eq. (10), the maximum ($E_f^{\max}$), midpoint (E_f^{mid}) and minimum ($E_f^{\min}$) values of critical resistant energy (E_f) of micro plastic deformation to generate the crack can be estimated of borosilicate glasses. Figure 8 shows changes in the $E_f^{\max}$, E_f^{mid} and $E_f^{\min}$ values against irradiation dose. Although HIEBL at 0.04 MGy slightly decreases the $E_f^{\max}$ value, it increases the E_f^{mid} and $E_f^{\min}$ values. Namely, 0.04 MGy-HIEBL tremendously decreases the experimental errors of E_f values. It is concluded that 0.04 MGy-HIEBL homogenizes the borosilicate glass.

The maximum values of $E_f^{\max}$ and $E_f^{\min}$ values are found at 0.22 and 0.04 MGy of irradiation dose, respectively. The high values of E_f^{mid} are obtained from 0.04 to 0.22 MGy of irradiation dose.

On the other hand, HIEBL from 0.22 to 0.43 MGy apparently decreases the $E_f^{\max}$ and E_f^{mid} values. HIEBL from 0.04 to 0.43 MGy also decreases the $E_f^{\min}$ values, although they are more than that before irradiation (broke line in Fig. 8).

4.2 Critical resistant energy of micro-plastic deformation to propagate the crack (E_f^p)

The fracture toughness (K_{IC}) is estimated by fracture

Fig. 8 Changes in the maximum ($E_f^{\max}$), midpoint (E_f^{mid}) and minimum ($E_f^{\min}$) values of critical resistant energy (E_f) of micro plastic deformation to generate the crack of borosilicate glass against EB irradiation dose. Broken line corresponds to the critical resistant energy of micro-plastic deformation to propagate the crack (E_f^p).

toughness to propagate the crack using standard indentation fracture (see eq. (5)). As shown in Fig. 3, the K_{IC} value of the borosilicate glass doesn't depend on the irradiation dose. If EB-irradiation annihilated the spontaneous crack origins, the minimum E_f value ($E_f^{\min}$) before irradiation was the critical resistant energy of micro-plastic deformation to propagate the crack (E_f^p). Therefore, the E_f^p value of the borosilicate glass doesn't depend on the irradiation dose, as shown in Fig. 8 (see broken line).

4.3 Critical resistant energy of micro-plastic deformation to generate the crack (E_f^n)

If the critical resistant energy of micro-plastic deformation to generate (nucleate) the crack (E_f^n) is defined, it expressed by a following equation.

$$E_f^n = E_f - E_f^p \quad (11)$$

When the crack origin is free in the glass, it is possible that the E_f value should be the $E_f^{\max}$ value. Thus, the ideal critical resistant energy of micro-plastic deformation to generate (nucleate) the crack ($^{\text{ideal}}E_f^n$) can be estimated by a following equation.

$$^{\text{ideal}}E_f^n = E_f^{\max} - E_f^p \quad (12)$$

If the ideal value ($^{\text{ideal}}E_f^n$) is the deference between $E_f^{\max}$ and E_f^p values, the irradiation dose dependence of $^{\text{ideal}}E_f^n$ values are shown in Fig. 9. HIEBL of less than 0.22 MGy increases the $^{\text{ideal}}E_f^n$ value, whereas HIEBL from more than 0.22 to less than 0.43 MGy decreases the $^{\text{ideal}}E_f^n$ values. The $^{\text{ideal}}E_f^n$ value at 0.43 MGy is lower than that before treatment.

The $^{\text{ideal}}E_f^n$ value of borosilicate glass irradiated (less than 0.22 MGy) is always larger than the E_f^p value. The $^{\text{ideal}}E_f^n$ value of borosilicate glass before irradiation is about nine times larger than the E_f^p value. HIEBL less than 0.22 MGy increases the $^{\text{ideal}}E_f^n$ value. As a result, the $^{\text{ideal}}E_f^n$ value of

Fig. 9 Changes in the ideal critical resistant energy of micro-plastic deformation to nucleate the crack (E_f^n) of borosilicate glass against EB irradiation dose. Broken line approximately corresponds to the critical resistant energy of micro-plastic deformation to propagate the crack (E_f^p).

borosilicate glass irradiated at 0.22 MGy is about 16 times larger than the E_f^p value.

As a conclusion, homogeneous irradiation of electron beam with low potential (HIEBL) mainly prevents to generate (nucleate) the crack, although HIEBL doesn't largely affect the resistance to crack propagation.

4.4 Controlling crack generation by dangling bonds

Annealed borosilicate glass is brittle because of the sharp potential curve of tight network structure with high atomic density. The sharp potential curve exhibits the strong inter-atomic force of ionic bonding at the minimum value of the curve, whereas the atomic scale plastic deformation induces the weak inter-atomic force of ionic bonding with long inter-atomic distance.

In order to improve the fracture strain, relaxation sites have been introduced by using HIEBL. It provides the 796, 805, 252, 507 kJ/mol. (here, based on the atomic mass (19.56 g/mol) of borosilicate glass, 1 kJ/mol-irradiation energy corresponds to 5.11×10^{-2} MGy ($= 51.1$ J/g)-irradiation dose.) of energy that is necessary to break chemical bonds between Si-O, B-O, Na-O and Al-O pairs.²⁴⁾

Based on the increasing the height of electron spin resonance (ESR) spectra, HIEBL not only reforms the crack tip from sharp to dull, but also easily forms dangling bonds at Na-O pairs in silica glassy network structure. Since HIEBL results in the formation of dangling bonds from Na-O pairs, this should result in a partial relaxation of residual molecular strains in the network structure. As a result, it can be explained that HIEBL from 0.04 to 0.22 MGy enhances the critical load (P_f) and critical diagonal (d_f) of micro plastic deformation to generate the crack (see Figs. 5 and 6).

When the interatomic distance of the bonded Si-O pair is optimal in the interatomic potential curve, relaxation increases the bonding energy of the network structure. Therefore, it is concluded that the relaxation of the Si-O network contributes to an increase in the critical resistant energy of micro-plastic deformation to generate (nucleate) the crack

(E_f^n), resulting in E_f enhancement, as shown in Figs. 8 and 9. Namely, the increased E_f value is also due to the relaxation of the network structure, as well as the annihilation of crack origin. Therefore, HIEBL prevents the crack generation (nucleation).

On the other hand, an additional irradiation dose of more than 0.22 MGy (kJ/g) causes an apparent decrease in the E_f^n (see Fig. 9) and E_f values (see Fig. 8). HIEBL of more than 0.22 MGy (kJ/g) apparently decreases the critical load (P_f) and critical diagonal (d_f) of micro plastic deformation to generate the crack (see Fig. 6). The additional irradiation at 0.43 MGy (kJ/g) increases the additional density of dangling bonds in the Si-O network structure (see Fig. 7). If the additional density of dangling bonds partly corresponds to the crack origins, the deterioration in the E_f^n (see Fig. 9) and E_f values (see Fig. 8) of samples irradiated from 0.22 to 0.43 MGy (kJ/g) can be explained.

5. Conclusion

An effect of homogeneous irradiation of electron beam with low potential (HIEBL) on the micro-fracture resistance of borosilicate glass was studied by using indentation of hardness test.

(1) HIEBL of less than 0.43 MGy didn't affect the fracture toughness (K_{IC}), which was estimated by fracture toughness to propagate the crack using standard indentation fracture method.

(2) HIEBL of less than 0.22 MGy increased the critical load (P_f), critical diagonal (d_f) and critical energy (E_f) of micro plastic deformation to generate the crack.

(3) Since 0.04 MGy-HIEBL tremendously decreased the experimental errors of E_f values, 0.04 MGy-HIEBL homogenized the borosilicate glass.

(4) The high values of the midpoints (E_f^{mid}) of E_f values were obtained from 0.04 to 0.22 MGy of irradiation dose.

(5) If EB-irradiation annihilated the spontaneous crack origins, the minimum E_f value (E_f^{min}) before irradiation was the critical resistant energy of micro-plastic deformation to propagate the crack (E_f^p). Thus, the E_f^p value of the borosilicate glass didn't depend on the irradiation dose.

(6) The critical resistant energy of micro-plastic deformation to generate the crack (E_f^n ; $E_f^n = E_f - E_f^p$) was defined. HIEBL increased the E_f values because of increasing E_f^n values.

(7) Based on the increasing the height of electron spin resonance (ESR) spectra, HIEBL formed dangling bonds in silica glassy network structure, as well as reformed the crack tip from sharp to dull. Thus, they probably relaxed the residual strain. If the partial relaxation of the residual strain mainly occurred around these dangling bonds of the sodium (Na)-oxygen pairs in the network structure, the increased E_f value was mainly due to the relaxation of the network structure, as well as the annihilation of crack origin. Therefore, HIEBL prevented the crack generation.

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