Tensile Properties of Optical Fiber Irradiated by Low Voltage Electron Beam Homogeneously

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1. Introduction

Since optical fibers (OF) simultaneously transfer communication signals over long distances with small loss of light transfer without influence of electromagnetic noise, they have been utilized for stabilized telecommunication systems.1,2 Extra-fine optical fiber for submarine cable is constructed with core and clad of silica glass covered with polymer, as shown in Fig. 1. Additions of Ge and P into silica core raises reflective index, whereas B and F additions into silica clad reduces the index. When the submarine cable is set in the ocean, the sites and tensile stress have been carefully determined by precise computer controlling. However brittleness is a serious problem that takes time and cost for repairing the submarine cables. Thus, strengthening the OF is always expected.

The electron beam irradiation with extremely low voltage of less than a 0.30 MeV-class simply charges materials, whereas the focusing electron beam irradiation with more than one MeV-class high voltage easily induces the radiation damage.4) Radiation damage has been observed in carbon fiber reinforced polymer (CFRP) irradiated by electron beam with high voltage of 1.5 MeV and high absorbed dose of 10 GGy (10⁴ J m⁻²).5)

On the contrary, the impact values of the both CFRP and glass fiber reinforced polymer (GFRP) have been improved by homogeneous low voltage 0.10 MeV class electron beam irradiation (HLEBI).6,7 The additional strengthening by HLEBI can be mostly explained by not only the fracture strain and stiffness of polymers,9,10 but also impact values of ceramics.11-14 Furthermore, it can be partly explained that the interface strengthening is subjected to surface activation induced by charging, as well as compressive stress at interface by dangling bond formation in polymer and fibers in CFRP and GFRP. In order to reduce the total cost and time for repairing the submarine cables during construction, the enhancements of elasticity, fracture stress and fracture strain have been expected. The elasticity can be estimated by the slope (dσ/dε) of stress-strain curves. The purpose of the present work is to investigate effects of HLEBI on dσ/dε, tensile strength and fracture strain of optical fiber (OF).

2. Experimental Procedure

2.1 Optical fiber and tensile test

Figure 1 show a schematic cross section of optical fiber (0.125 mm radius) utilized for submarine cable. It was constructed with core (0.0100 mm diameter), clad (0.125 mm diameter outside) and ultraviolet cured (UV) resin of acryl-urethane sheath (62.5 µm thickness) (Fujikura Ltd., SM.10.UV). The core and clad were made from SiO₂, Ge and Yuu Nakahara2

(Received November 16, 2011; Accepted April 13, 2012; Published May 30, 2012)

Keywords: optical fiber, optical fiber, tensile test, low voltage electron beam homogeneously

\[ \sigma = F/S \] (1)

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Here, \( \sigma, F \) and \( S \) are tensile stress (MPa), tensile load (N) and cross section area (m\(^2\)), respectively. The stiffness was evaluated by the slope \((d\sigma/de)\) at each strain in stress–strain curves. The distance between supporting points was 60.0 mm.

### 2.2 Condition of HLEBI

Sheet electron beam irradiation with low energy was homogeneously performed using an electron-curtain processor (Eye electron beam Co. Ltd., Type CB250/30/20mA).\(^{6-14}\) The specimen was machined from the sheet heat-treated for making composites and was homogeneously irradiated with the electron beam through a titanium thin film window attached to a vacuum chamber, 240 mm in diameter. Since HLEBI treatment was after forming interface between silica clad and acryl-urethane sheath, improvement of adhesive force could be expected. A tungsten filament in a vacuum was used to generate the electron beam at a low energy (acceleration potential, \( V \); MeV), of 0.170 MeV and irradiating current density \((J; A \text{ m}^-2)\) of 0.089 A m\(^{-2}\). Although electron beam generation was done in a vacuum, the irradiated sample was kept under protective nitrogen at atmospheric pressure. The distance between sample and window was 35 mm. To prevent oxidation, the samples were kept in a protective one atmosphere of nitrogen gas with a residual concentration of oxygen below 0.003%. The flow rate of nitrogen gas was 1.5 L s\(^{-1}\) at 0.100 MPa of nitrogen gas pressure. Each absorbed 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 m \( \times \) 0.15 m) was transported on a conveyor at a speed of 10 m min\(^{-1}\). The sheet electron beam irradiation was applied intermittently. Repeated irradiations to both side surfaces of samples were used to increase the total dose of irradiation. The interval between the end of one period of irradiation and the start of the next operation was 30 s. When the irradiation current \((J; \text{mA})\), the conveyor speed \((v; \text{m min}^{-1})\) and number of irradiations \((N)\) are determined, the irradiated dose \((D; \text{MGy})\) is expressed by a following equation.

\[
D = 0.216(J/v)N
\]  

The absorbed dose was controlled by the integrated irradiation time in each of the samples. Here, absorbed dose was corrected by using FWT nylon dosimeter of RCD radiometer film (FWT-60-00: Far West Technology, Inc. 330-D South Kellogg Goleta, California 93117, USA) with irradiation reader (FWT-92D: Far West Technology, Inc. 330-D South Kellogg Goleta, California 93117, USA). The dose was 0.043 MGy at each irradiation.

Penetration depth of the electron beam is one of the important factors for dominating productivity and CFRTP thickness choice to apply for practical use. Based on the density \((\rho; \text{kg m}^{-3})\) and irradiation voltage \((E; \text{MeV})\), the EB-irradiation depth \((D_\text{HE})\) is expressed by the following equation proposed by Christenhusz and Reimer.\(^{15}\)

\[
D_\text{HE} = 66.7 \sqrt{3/\rho}
\]  

The surface electrical potential (0.13 MeV) is estimated from the electrical potential (0.17 MeV), the 10 µm thickness of the titanium \((\rho = 4.5 \text{ Mg m}^{-3})\) window and the 30 mm distance between the sample and the window in the nitrogen gas atmosphere \((\rho = 1.1 \text{ kg m}^{-3})\).

When the densities of the glass fiber (core and cladding) and UV-resin (acryl-urethane sheath) are assumed to be those of silica \((2100 \text{ kg m}^{-3})\) and polyurethane (PU; 1130 kg m\(^{-3}\)), the effective EB-irradiation depth values are calculated to be 100 and 192 µm, respectively.

Another method proposed by Libby relates it to the mass thickness \((l_0; \text{g m}^{-2})\) and irradiation voltage \((E_\text{irr}; \text{kV})\), expressed by the following equation.\(^{16}\)

\[
l_0 = E_\text{irr}^{5/3}/150
\]  

The estimated mass thickness is 34.8 g m\(^{-2}\), when the initial irradiation voltage is 170 kV. Since both mass thickness values of Ti foil \((17.8 \text{ g m}^{-2})\) and N\(_2\) gas \((1.50 \text{ g m}^{-2})\) reduced the EB-irradiation depth, the mass thickness of sample was 34.8 g m\(^{-2}\).

The effective penetration depth values of HLEBI for silica glass and PU were estimated as 100 and 192 µm by the first method, whereas they were 146 and 286 µm by the second, respectively. The calculated values were concerning only the irradiation from one surface. The irradiation had been carried out on both surfaces here. Using either calculation,\(^{15,16}\) it is clear that the irradiation had been totally penetrated though the 62.5 µm thick coating UV-resin sheath from surface and silica glass from interface. Since the irradiated depth from sample surface was almost 100% of the 250 µm diameter optical fiber, it is expected that the HLEBI improved the mechanical properties.

### 2.3 ESR observation

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 spectrometer (ESR, JES-FA200, JEOL Ltd., Tokyo).\(^{17,18}\) The microwave frequency range used in the ESR analysis was the X-band at 9.5 ± 0.050 GHz with a field modulation of 0.10 MHz. The microwave power was 1.0 mW. The magnetic field was varied from 0.32 to 0.33 T. The spin density was calculated by using the Mn\(^{2+}\) standard sample. Only ESR spectra were given, not spin densities. Based on the standard calibration material \([4\text{-hydroxy-2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPOL, 0.089-04191, Wako Pure Chemical Industries Ltd., Tokyo)]\) and Mn\(^{2+}\) in the MnO, the density of dangling bonds was estimated by double integration of the intensity of the ESR signal.\(^{18}\)
3. Results

3.1 Stress–strain curve before and after HLEBI

Evaluating the cumulative probability of tensile elastic modulus ($P_E$) (fracture probability ($P_f$)) is a convenient method of quantitatively analyzing experiment values. $P_E$ ($P_f$) are expressed by the following equation, which is a generalized form of the median rank method.\(^{[19]}\)

$$P_E (P_f) = (i - 0.3) / (N_i + 0.4)$$  \(\quad\) (5)

Here $N_i$ and $i$ are the total number of samples ($N_i = 11$) and the order of fracture of each sample, respectively, where the orders of both fracture strength and fractured strain are the aligned number of fractured samples from low to high value. When the $i$ value is 6, the $P_E$ ($P_f$) values is 0.50.

Figure 3 shows tensile stress–strain curves at mid $P_E$ of 0.5 of the submarine optical fiber (OF) before and after HLEBI of 0.65 and 1.04 MGy. HLEBI apparently raises tensile strength of fracture stress ($\sigma_f$) and fracture strain ($\varepsilon_f$), although remarkable effect of HLEBI on the slope ($d\sigma/d\varepsilon$) of OF can not be observed in Fig. 4.

3.2 Elasticity evaluated by ($d\sigma/d\varepsilon$)$_{\text{max}}$ against cumulative probability ($P_E$)

In order to clarify the changes in the slope ($d\sigma/d\varepsilon$) values of stress–strain curves at mid $P_E$ of 0.5 of the optical fiber before and after HLEBI of 0.65 to 1.04 MGy, a relationship between $d\sigma/d\varepsilon$ and tensile strain ($\varepsilon$) are illustrated. Figure 4 shows changes in $d\sigma/d\varepsilon$ against $\varepsilon$. The initial slope value ($d\sigma/d\varepsilon|_0$) has been obtained when $\varepsilon$ is zero. The ($d\sigma/d\varepsilon$)$_0$ value strongly corresponds to tensile elastic modulus. Figure 5 shows changes in ($d\sigma/d\varepsilon$)$_0$ of OF treated by each dose of HLEBI against $P_E$ value. HLEBI from 0.30 to 1.17 MGy apparently enhances the ($d\sigma/d\varepsilon$)$_0$ value of the submarine optical fiber, although the effect of slight dose of HLEBI from 0.043 to 0.22 MGy on ($d\sigma/d\varepsilon$)$_0$ cannot be found.

The maximum value of ($d\sigma/d\varepsilon$) corresponds to ($d\sigma/d\varepsilon$)$_{\text{max}}$, as shown in Fig. 4. The maximum slope value ($d\sigma/d\varepsilon|_{\text{max}}$) has been obtained. Figure 6 shows changes in ($d\sigma/d\varepsilon$)$_{\text{max}}$ of submarine optical fiber irradiated at each dose.

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**Fig. 3** Stress–strain curves at mid $P_E$ of 0.5 of submarine optical fiber before and after HLEBI of 0.65 and 1.04 MGy.

**Fig. 4** Changes in ($d\sigma/d\varepsilon$) values of stress–strain curves at mid $P_E$ of 0.5 against tensile strain ($\varepsilon$) of optical fiber before and after HLEBI of 0.65 and 1.04 MGy.

**Fig. 5** Changes in ($d\sigma/d\varepsilon$)$_0$ against each cumulative probability of tensile elastic modulus ($P_E$) of submarine optical fiber irradiated at each dose.

**Fig. 6** Changes in ($d\sigma/d\varepsilon$)$_{\text{max}}$ against each cumulative probability of tensile elastic modulus ($P_E$) of submarine optical fiber irradiated at each dose.
OF treated by each dose of HLEBI against $P_E$ value. HLEBI from 0.30 to 1.17 MGy mostly enhances the $(d\sigma/d\varepsilon)_\text{max}$ value of the submarine optical fiber, although the effects of slight dose of HLEBI from 0.043 to 0.22 MGy on $(d\sigma/d\varepsilon)_\text{max}$ cannot be found.

Consequently, HLEBI slightly and drastically enhances $(d\sigma/d\varepsilon)_i$ and $(d\sigma/d\varepsilon)_\text{max}$.

4. Discussion

4.1 Effect of HLEBI on $(d\sigma/d\varepsilon)_i$ and $(d\sigma/d\varepsilon)_\text{max}$

Figure 7 shows changes in $(d\sigma/d\varepsilon)_i$ and $(d\sigma/d\varepsilon)_\text{max}$ at mid $P_E$ of the submarine optical fiber against dose of HLEBI dose. $(d\sigma/d\varepsilon)_\text{max}$ is always higher than $(d\sigma/d\varepsilon)_i$.

HLEBI from 0.39 to 1.17 MGy apparently enhances the $(d\sigma/d\varepsilon)_i$ at mid $P_E$. The highest $(d\sigma/d\varepsilon)_i$ value of 5.3 GPa, which is about 10% higher than that (4.8 GPa) before irradiation, is found at 1.04 MGy. In addition, the slight drop of $(d\sigma/d\varepsilon)_i$ at mid $P_E$ is found in OF treated by additional HLEBI dose of 1.17 MGy, although the $(d\sigma/d\varepsilon)_i$ value is higher than that before HLEBI.

On the other hand, HLEBI from 0.30 to 1.17 MGy also enhances the $(d\sigma/d\varepsilon)_\text{max}$ at mid $P_E$. The highest $(d\sigma/d\varepsilon)_\text{max}$ value of 5.9 GPa, which is about 20% higher than that (4.9 GPa) before irradiation, is found at 1.04 MGy. In addition, the drop of $(d\sigma/d\varepsilon)_\text{max}$ at mid $P_E$ is found in OF treated by additional HLEBI dose of 1.17 MGy, although the $(d\sigma/d\varepsilon)_\text{max}$ value is higher than that before HLEBI.

4.2 Effects of dangling bonds induced by HLEBI on strengthening of optical fiber

The $(d\sigma/d\varepsilon)_i$ and $(d\sigma/d\varepsilon)_\text{max}$ improvements of submarine optical fiber treated by HLEBI are mainly caused by increasing not only the elasticity enhancements of both silica glass and acryl-urethane, but also adhesive force at interface. The elasticity enhancements are probably caused by homogeneous atomic scale internal compressive stress induced by repulsive force between dangling bonds in silica core glass and acryl-urethane sheath.

In addition, the interfacial adhesive force is probably caused by attractive intermolecular force between terminated atoms with dangling bonds of the one side materials and terminated atoms without dangling bonds of the another side materials at the interface.

From the conventional X-ray diffraction patterns of the silica glass before and after the HLEBI, a remarkable difference cannot be observed. On the other hand, HLEBI in fact produces detectable dangling bonds. The ESR signals corresponding to dangling bonds of polymer with and without HLEBI are generally observed. HLEBI generates the dangling bonds for silica glass and enhances its density. When HLEBI raises the density up to the optimum value, the impact values improvement and strengthening have been found in the silica glass and polymer, respectively.

When HLEBI cuts the weak chemical bonds in both silica and polymer, the repulsive force between terminated atoms mostly occurs at each dangling bond, resulting in generating volume expansion. Based on the radial distribution function of X-ray of silica glass, the formation of dangling bonds generated by HLEBI enhances the mean atomic distance and reduces the coordination number, resulting in volume expansion. The volume expansion induced by HLEBI probably generates the atomic scale internal compressive stress to enhance the $(d\sigma/d\varepsilon)_i$ and $(d\sigma/d\varepsilon)_\text{max}$ in silica and polymer.

To discuss the effects of HLEBI on the $(d\sigma/d\varepsilon)_i$ and $(d\sigma/d\varepsilon)_\text{max}$ of submarine optical fiber (OF), ESR signals related to dangling bonds have been observed. Figure 8 exhibits the ESR signals of OF before and after HLEBI, together with silica glass. The ESR signals related to dangling bonds can not be found in silica glass before irradiation, whereas a small signal is found in OF before HLEBI. Furthermore, the small dose of 0.04 MGy-HLEBI generates the sharp ESR signals in OF. Moreover, increasing HLEBI dose from 0.04 to 1.04 MGy raises the intensity values of ESR signals strongly related to the density of dangling bonds in OF.

4.3 Fracture stress ($\sigma_f$) and fracture strain ($\varepsilon_f$)

Figures 9 and 10 show changes in $\sigma_f$ and $\varepsilon_f$ of OF against fracture probability ($P_f$) value at each dose of HLEBI. Remarkable effects of HLEBI of 0.65 MGy on $\sigma_f$ and $\varepsilon_f$ of OF have been obtained at each $P_f$ value. Although the additional EB irradiation of 1.04 MGy mostly reduces the $\varepsilon_f$ at $P_f$ under 0.8, it is increased at high $P_f$ of more than 0.8, and the HLEBI of 1.04 MGy also remarkably enhances the $\sigma_f$ value of the OF. Thus, 0.65 MGy-HLEBI mostly enhances the $\sigma_f$ and $\varepsilon_f$ of the submarine optical fiber without remarkable radiation damage.

4.4 Effects of dangling bonds induced by HLEBI on interfacial friction force of submarine optical fiber

When the atomic scale internal compressive stress induced by dangling bond formation enhances the friction force at interface between silica glass clad and acryl-urethane, it is able to prevent to pull out the silica glass fiber from the acryl-urethane matrix. Since HLEBI also enhances the density of dangling bonds of glass fiber and acryl-urethane sheath of OF, effects of compressive stress on pull-out resistance and elasticity enhancements probably occur.
Thus, we conclude that the interfacial friction force as well as the strengthening of both glass fiber and acryl-urethane probably contributes to the HLEBI effects to enhance the $(d\sigma/d\epsilon)_i$, $(d\sigma/d\epsilon)_{\text{max}}$, $\epsilon_f$, and $\sigma_f$ values of OF.

As shown in Fig. 10, the additional EB irradiation of 1.17 MGy apparently reduces the $\epsilon_f$, and improves the $\sigma_f$. Since the additional irradiation easily breaks the chemical bonds of acryl-urethane and glass fiber, it probably generates excess fracture sites for crack origins and its propagation. The decay of $\epsilon_f$ of OF irradiated at 1.04 MGy can be explained by the excess formation of dangling bonds.

Therefore, it can be explained that 0.64 MGy-HLEBI enhances the $(d\sigma/d\epsilon)_i$ and $(d\sigma/d\epsilon)_{\text{max}}$ as well as $\epsilon_f$, resulting in enhancement of $\sigma_f$ of OF.

4.5 Compressive stress and attractive intermolecular force induced by dangling bonds generated by HLEBI

When dangling bonds form at terminated atoms in polymers, it is possible to generate the compressive stress by thermal expansion at both insides of silica glass fiber and acryl-urethane sheath and their interface. When the compressive stress is the dominant factor, it prevents to generate and propagate the crack. The compressive stress should also enhance the fracture strain, elasticity and resistivity to plastic deformation, as well as kinetic and static friction force. They should enhance the tensile strength.

In addition, the interfacial adhesive force is probably caused by attractive intermolecular force between terminated atoms with dangling bonds of the one side materials and terminated atoms without dangling bonds of the another side materials at the interface. When the attractive intermolecular force is the other dominant factor, it should enhance the static friction apparently higher than the kinetic friction force. Thus, it enhances the elasticity, whereas it reduces the fracture strain.

Based on the results in Figs. 7, 9 and 10, 0.65 MGy-HLEBI enhances the $(d\sigma/d\epsilon)_i$, $(d\sigma/d\epsilon)_{\text{max}}$, $\epsilon_f$, and $\sigma_f$. Therefore, the dominant factor of the impact value of the sample treated by 0.65 MGy-HLEBI is the compressive stress induced by dangling bonds formation.

On the other hand, 1.04 MGy-HLEBI enhances the $\sigma_f$ (see Fig. 9) as well as tremendously enhances the $(d\sigma/d\epsilon)_i$ and $(d\sigma/d\epsilon)_{\text{max}}$ (see Fig. 7), although it reduces the $\epsilon_f$.
(see Fig. 10). Thus, the dominant factor of the impact value of the sample treated by 1.04 MGy-HLEBI is the attractive intermolecular force induced by dangling bonds formation.

5. Conclusion

In summary, homogeneous low voltage electron beam irradiation (HLEBI) improved the elasticity indicated by the initial and maximum slope value \((\sigma / \varepsilon)_i\) and \((\sigma / \varepsilon)_{\text{max}}\) of tensile test of optical fiber (OF) for submarine cable with 250 µm diameter. It was constructed with both core (10 µm diameter) and clad silica (125 µm diameter) glasses inside, and acryl-urethane sheath (62.5 µm thickness).

1. HLEBI at 1.17 MGy raised tensile strength of fracture stress \((\sigma_f)\) and its strain \((\varepsilon_f)\), as well as \((\sigma / \varepsilon)_i\) and \((\sigma / \varepsilon)_{\text{max}}\).
2. HLEBI from 0.30 to 1.17 MGy apparently enhanced the \((\sigma / \varepsilon)_i\). The highest \((\sigma / \varepsilon)_i\) value of 5.3 GPa, which was about 10% higher than that (4.8 GPa) before irradiation, was found at 0.104 MGy.
3. HLEBI from 0.30 to 1.17 MGy also enhanced the \((\sigma / \varepsilon)_{\text{max}}\). The highest \((\sigma / \varepsilon)_{\text{max}}\) value of 5.9 GPa, which was about 20% higher than that (4.9 GPa) before irradiation, was found at 1.04 MGy.
4. Remarkable improvements of HLEBI of 0.65 MGy on tensile strength \((\sigma_f)\) and fracture strain \((\varepsilon_f)\) of OF were obtained at each fracture probability \((P_f)\) value. Although the additional EB irradiation of 1.04 MGy mostly reduced the \(\varepsilon_f\), it improved the \(\sigma_f\).
5. Since HLEBI also enhanced the density of dangling bonds of each material of OF, effects of compressive stress on pull-out resistance and elasticity enhancements of silica fiber inside and acryl-urethane sheath probably occurred. Thus, 0.64 MGy-HLEBI enhanced the \((\sigma / \varepsilon)_i\) and \((\sigma / \varepsilon)_{\text{max}}\) as well as \(\varepsilon_f\) resulting in enhancement of \(\sigma_f\) of OF.

Acknowledgement

Authors would like to thank Dr. Keisuke Iwata and Mr. Keisuke Takada, Prof. Michael C. Faudree and Prof. Dr. Akira Tonegawa of Tokai University for their useful help.

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