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Effects of quenching, irradiation, and annealing processes on the radiation hardness of silica fiber cladding materials (I)

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ABSTRACT

Silica optical fiber cladding materials were experimentally treated by a series of processes. The treatments involved quenching, irradiation, followed by annealing and subsequent re-irradiation, and they were conducted in order to improve the radiation hardness. The microstructural properties of the treated materials were subsequently investigated. Following the treatment of the optical fiber cladding materials, the results from the electron spin resonance (ESR) analysis demonstrated that there was a significant decrease in the radiation-induced defect structures. The ESR signals became significantly weaker when the samples were annealed at 1000 °C in combination with re-irradiation. In addition, the microstructure changes within the silica optical fiber cladding material were also analyzed using Raman spectroscopy. The experimental results demonstrate that the Si—O—Si bending vibrations at ω_3 = 800–820 cm^{-1} and ω_4 = 1000–1200 cm^{-1} (with longitudinal optical (LO) and transverse optical (TO) splitting bands) were relatively unaffected by the quenching, irradiation, and annealing treatments. In particular, the annealing process resulted in the disappearance of the defect centers; however, the LO and TO modes at the ω_3 and ω_4 bands were relatively unchanged. With the additional support of the ESR test results, we can conclude that the combined treatment processes can significantly enhance the radiation hardness properties of the optical fiber cladding materials.

1. Introduction

Free-water peak single-mode optical fiber (FWP-SMF) has become widely used to extend the bandwidth of Dense Wavelength Division Multiplexing (DWDM) communication systems as a result of its free-water absorption at 1380 nm band [1,2]. With the continuous development of optical fiber applications in the radiation fields, such as nuclear power plants, nuclear waste treatment facilities, and space missions, the irradiation properties of optical fibers have attracted much research attention [3–10]. In recent years, areas of study have increasingly focused on the influence of irradiation on the optical properties of silica materials. The applications of silica materials include: optical transmission components in a fusion reactor environment, future development of nuclear facilities, and the development of new security or monitoring systems with improved resistance to harsh environments [3–6]. Research on microstructural properties of the new type of optical fibers is very important to enhance the radiation hardness performance.

Previous works have reported on the influences of irradiation on the various types of optical fibers [11–16] and on the calculation models of silicon dioxide microstructures [17–21]. However, the influence of irradiation on the network structure of optical fiber cladding materials is not yet clear. Additionally there has been limited research on the use of silica fiber material treatments process for the improvement of radiation hardness properties.

In this paper, we investigate the influences of a series of processes on the microstructural properties of the optical cladding material. The process involved quenching in combination with irradiation, following by annealing, and subsequent re-irradiation. The microstructure was analyzed by both ESR and Raman spectroscopy. These treatment methods can improve the radiation hardness properties of the silica optical fiber cladding material.

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2. Materials and experimental

Two experimental samples, Nos. 1 and 2, were obtained from the cladding part of a free-water single mode optical fiber (ITU-T G.652D) preform (Jiangsu Fasten Photonics Co., Ltd. Jiangsu, China), which constituted of pure amorphous silica (a-SiO_2). The typical dimensions of the samples are $8 \times 3 \times 2$ mm ($L \times W \times H$). The weight was approximately 0.11 g. The experimental procedure was as follows: firstly, the samples were quenched at 1300 and 1400 °C. They were subsequently irradiated by a 10 kGy doses at room temperature with γ-rays from a ^{60}Co radiation source (Radiation Center at Shanghai Academy of Agricultural Sciences, China). The dose rate was 800 Gy/h. Secondly, the samples were annealed at 600 and 1000 °C, and thermally stabilized for 30 min. They were subsequently air cooled to room temperature. Finally, the fiber cladding materials were treated by re-irradiation with a dose of 10 kGy.

The ESR spectra of the above samples were recorded at the X band on a JES-FA200 Electron Spin Resonance Spectrometer (Shanghai Institute of Applied Physics, Chinese Academy of Sciences). The modulation frequency was 100 kHz, operating in the X-band frequency at 9.53 GHz, and the center magnetic field strength was 326 mT. The sweep range was 50 mT, the response time was 0.25 s, and the microwave power was 50 mW. All the ESR spectra were obtained at room temperature. The ESR measurement conditions were confirmed not to distort the ESR signal or induce microwave saturation effects.

The Raman spectra of the fiber cladding materials were measured by a Renishaw in Via plus a laser Raman spectrometer in the range of 200 cm^{-1} to 1000 cm^{-1} with a spectral resolution of 0.5 cm^{-1}. Each polished naked fiber sample was severed by an optical fiber cleaver, and the optical fiber cross section was excited by an argon ion laser. The Raman spectra were obtained from a cross-section of the surface, with a spot size of approximately 100 μm.

3. Experimental results and discussion

3.1. Quenching and irradiation processes

The two samples were quenched at 1300 and 1400 °C, and were subsequently irradiated with a dose of 10 kGy. The ESR signals spectra were measured, as shown in Fig. 1. The experimental

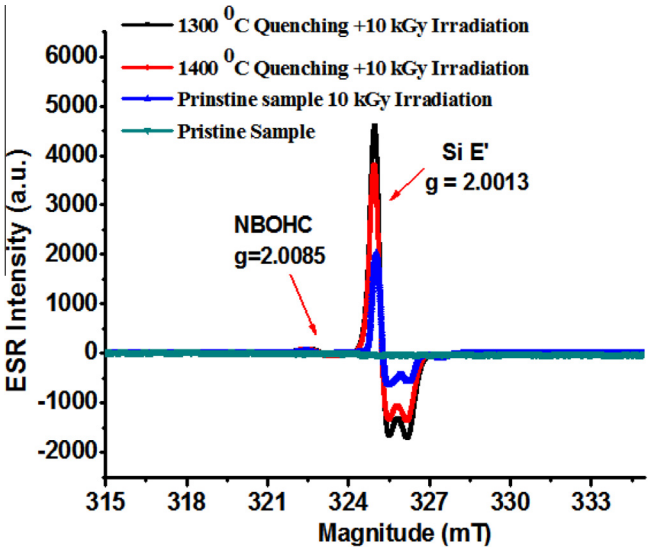

Fig. 1. ESR spectra of samples Nos. 1 and 2 following the various process treatments.

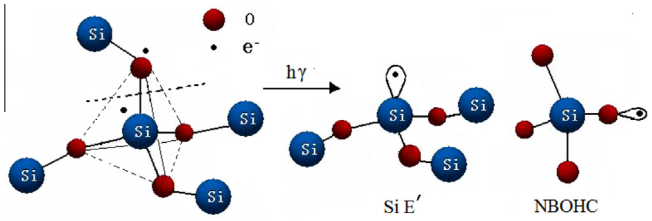

Fig. 2. Formation process of SiE' and NBOHC defect centers.

results demonstrate that there were no obvious signals of defect centers detected in the pristine sample. Following an irradiation dose of 10 kGy, there was a clear signal of the ESR spectrum, which indicates that color centers were generated. The ***g*** value (Landé *g*-factor) was 2.0085 and 2.0013, respectively, as indicated by the blue line in Fig. 1. It can be observed that $g = 2.0013$ lies around the E' center, characterized by *g* values: $g = 2.0018$ and $g = 2.001$, respectively [2,22,23]. Therefore, the ESR peak labeled $g = 2.0013$ is the result of the joint action of the E' defect centers. The signal labeled $g = 2.0085$ corresponds to the non-bridging oxygen hole center (NBOHC), characterized by the g values: $g = 2.0095$ [2,24]. Here, NBOHC is not easy obverse at room temperature. The SiE' and NBOHC in the fiber material are mainly derived from the Si—O—Si bond breakage [25,26], as shown in Fig. 2.

We can also see from Fig. 1, that following the irradiation of the unquenched fiber samples (with a dose of 10 kGy), there is a slight increase in the signal intensity of ESR. Following the quenching at 1300 and 1400 °C in combination with the 10 kGy irradiation, respectively, there is a significant increase in the signal intensity of the ESR spectra. There is a greater increase in the signal intensity of the ESR from samples that were quenched at 1300 °C in comparison to those quenched at 1400 °C. The quenching process may change the microstructures of the fiber cladding materials which induced the increase of ESR signal intensity.

3.2. Annealing and re-irradiation processes

The thermal annealing treatment was used to repair the defect structures. Samples Nos. 1 and 2 were irradiated with a dose of 10 kGy, subsequently heated up to 1000 °C, and thermally stabilized for 30 min. The samples were then annealed to room temperature by air cooling. The ESR signals are shown in Fig. 3. It is clear that all defect centers of the SiE' and NBOHC have completely disappeared, which implies that the high-temperature thermal process can annihilate the radiation-induced defect structures [2,22]. Specifically, the broken Si-O bonds in the fiber cladding material are reconnected to form a stable structure through the thermal annealing process.

Following the series of treatment processes, the fiber materials are re-irradiated with 10 kGy. The ESR signals demonstrate various changes, as shown in Figs. 4 and 5. Following the re-irradiation, there is a slight increase in the defect centers of SiE' and NBOHC. This can be particularly observed after the quenching at 1400 °C in combination with irradiation. In combination with the 1000 °C annealing treatments, the ESR signal is much weaker.

In addition, the microstructural changes in the silica optical fiber cladding material were also analyzed using Raman spectroscopy, as shown in Fig. 6. Six sharp peaks can be observed in the Raman spectra. The Peaks at $D_1 = 495$ and $D_2 = 606$ cm^{-1} correspond to the symmetrical stretching vibrations of the regular 4-MRs and the planar 3-MRs among the network structures, respectively [18,27]. The peak at $\omega_1 = 440$ cm^{-1} results from the Si—O—Si symmetrical stretching vibration [25]. The peak at $\omega_3 = 800$–818 cm^{-1} is attributed to the Si—O—Si bending vibration, and it is considered as two bands with longitudinal optical (LO) and