FISEVIER

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Temperature measurement by thermal luminescence of partially replaced core optical fiber

T. Shikama*, K. Toh, S. Nagata, B. Tsuchiya, Y. Ohno

Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan

ABSTRACT

A partially replaced core optical (PARCO) fiber – a fiber chip doped with high concentration of oxyhydrate (OH) inserted into a pure silica core fiber to measure temperatures with a spatial resolution. A heating experiment was performed at elevated temperatures with a PARCO fiber under gamma ray irradiation. The PARCO fiber had a characteristic thermal luminescent peak at 1390 nm that exhibited temperature dependence. When the temperature was raised up to 1000 °C, the other broad luminescence bands than the peak at 1390 nm was generated and grew. Optical surface observation showed that morphology change of a flat clad surface was taking place, suggesting some thermal damage of the surface. The damaged area had positive correlation with the intensity of the observed broad luminescence band. Transmission electron microscopy (TEM) showed that the damage related with the crystallization of the glassy structure of fused silica in the near-surface region.

© 2009 Elsevier B.V. All rights reserved.

1. Introduction

Optical sensors have several advantages over conventional electrical sensors for monitoring environmental parameters in nuclear systems. A temperature measurement is among one of the most important monitoring parameters in advanced nuclear systems where severe environmental conditions will cause entangled interferences with each other. The most conventional temperature measuring sensor is a thermocouple. But, it is well recognized that an electromotive force (EMF) of thermocouple is degraded by many radiation induced phenomena, such as radiation-enhanced diffusion at a two-legs junction, composition changes caused by nuclear transmutations. Radiation induced electromotive force (RIEMF) will be another source for disturbing a correct measurement of the EMF [1]. Recent improvement of radiation-resistance of fused silica (SiO₂) optical fibers (hereafter denoted as the optical fiber) is remarkable especially in the wavelength range of 800-1500 nm. Some fibers show a radiation induced optical transmission loss of less than a few dB/m, with a fast neutron fluence of 5×10^{24} n/m². Also, the radiation induced loss in this wavelength range is very flat as a function of a wavelength. Thus, optical measurements in this wavelength of 800-1500 nm are realistic even in heavy irradiation environments such as in a core region of nuclear power plants.

Intensity and spectrum of the black-body-radiation is the most popular temperature measuring method [2]. However, the wavelength region needed for the black-body-radiation is sometimes

* Corresponding author. E-mail address: shikama@imr.tohoku.ac.jp (T. Shikama). longer than 1500 nm, where optical transmissivity of the optical fiber is not excellent and it sometimes has strong wave-length dependence. Measuring the thermo-luminescence intensity of some luminous materials is another possibility. In the previous experiment, we demonstrated feasibility of temperature measurement by measuring thermo-luminescence of a single crystal alumina (Al₂O₃), namely a sapphire in a high flux fission reactor of Japan Materials Testing Reactor (JMTR) with a neutron flux larger than 5×10^{17} n/m² s up to a neutron fluence of about 5×10^{24} n/m², which will be corresponding to about a half year operation of conventional nuclear fission power reactor. However, it needs an appropriate optical coupling between thermo-luminescent material and an optical-signal transmitting optical fiber for realizing a reliable temperature measurement [3].

The optical fiber emits not only thermal optical radiation described by the Planck's radiation law but also strong thermal luminescence attributed to dopants in the optical fiber at elevated temperatures. The luminescent spectrum usually exhibits a characteristic peak depending on energy levels of the dopant [4]. Among impurities the oxyhydrate (OH) has a strong thermo-luminescence in the wavelength range of 800–1500 nm. The OH is the most popular dopant in the optical fiber and industrial technologies for controlling OH contents in the optical fiber is well established. The OH doped optical fiber itself could be used as a temperature sensor, but the obtained signal will yield an information of the highest temperature without giving us any information about the position. In the present study, we propose a partially replaced core optical (PARCO) fiber – a fiber chip doped with high OH content inserted into pure silica to realize spatial resolution. A heating experiment

^{0022-3115/\$ -} see front matter @ 2009 Elsevier B.V. All rights reserved. doi:10.1016/j.jnucmat.2008.12.204



Fig. 1. Schematic diagram of a partially replaced core optical (PARCO) fiber.

was performed at elevated temperatures with a PARCO fiber under gamma-ray irradiation.

2. Experimental

A schematic structure of the PARCO fiber is shown in Fig. 1. The optical fiber that has high content of OH is sandwiched between pure optical fibers, by connecting them with the arc fusion splicer (Fujikura Ltd.). The silica fiber of 800 ppm OH-doped with a length of 10 mm was sandwiched with the pure silica fiber. The main component of the core of the purified silica fiber was SiO₂ with low quantities of impurities or dopants such as OH and fluorine (F), while that of the clad was SiO₂ doped with F for lower optical reflective index. Impurities in the optical fiber tend to be precursors of radiation induced optical absorption, and purified optical fiber (the pure optical fiber) is one of candidates as a radiation resistant optical fiber in heavy irradiation environments. Diameter of the core/clad was 0.2/0.25 mm.

The gamma-ray irradiation was performed in the ⁶⁰Co gammaray irradiation facility at Tohoku University. The main energies of the ⁶⁰Co gamma-ray are 1.17 and 1.33 MeV, and they mainly cause electronic excitations through Compton scattering and inelastic collisions in optical fibers. Radioactivity of the gamma-ray source is 8.3 TBq, and the absorbed dose rate of the irradiated sample is 5.8 mGy/s. Detailed setup of the experiments can be found elsewhere [4]. The optical fiber was passed through the electrical heater. One end of the fiber was connected to an optical spectrum analyzer (Ando Electric, AQ-6315A) and the other was covered with aluminum foil. The optical detector can measure optical signals in the wavelength range from 400 to 1600 nm. The PARCO fiber with a length of 100 mm was in the electrical heater for the heating tests, and high-OH part of PARCO fiber was arranged at the center of the heater.

The heating rate was maintained at 3 °C/min.

3. Luminescence

An Arrhenius plot of measured optical intensity at 1390 nm is shown in Fig. 2. In the figure, filled circle indicates the measured optical intensities and the line indicates a calculated result from Arrhenius law. As shown in the inserted figure measured optical spectrum at 900 °C, the characteristic peaks at 1260 and 1390 nm were observed. The measured optical intensity showed good Arrhenius relation, and the value of the activation energy calculated from the slope of the line is 1.13 eV. The activation energy and the wavelengths of the peaks agreed with the values of the OH [5,6]. It is concluded that the luminescent peaks in Fig. 2 are coming from a thermally activated process of OH in the optical fiber. There is a possibility to use the thermal luminescent intensity of OH as a temperature measurement probe because the luminescent intensity has a temperature dependence.

To confirm a thermal stability of the observed luminescence peaks, the optical fiber was maintained at 900 °C for 60 h. The measured optical spectra as a function of the elapsed time, after the



Fig. 2. Arrhenius plot of measured intensity at 1390 nm. Inserted figure shows the measured optical spectrum at 900 $^\circ$ C.

temperature arrived at 900 °C, are shown in Fig. 3. It is confirmed that the spectral shape of the thermal luminescence did not change but the intensity of the peak decreased with the time. The change of the optical intensity was due to change of the content of the OH in the optical fiber [4,7]. In optical transmission measurements in infrared region, the optical fibers whose OH concentration is initially less than a few 10 ppm showed clear increase of the optical fiber of 800 ppm OH content showed the decrease of the OH concentration. It indicates that hydrogen is coming out or going into the optical fiber core and thermally equilibrium OH concentration with the air is below 800 ppm at 900 C. The incoming and outgoing of the hydrogen can be suppressed by a temperature-resistant jacket such as a refractory metal coating.

The stability of the OH luminescence was also examined at 1000 °C. Fig. 4 shows the measured optical spectra as a function of the elapsed time after the temperature reached at 1000 °C. Apparent different behaviors were observed, being compared with the results at 900 °C, namely, growth of wide band luminescence in the wavelength range above 800 nm (base-line luminescence),



Fig. 3. Spectra as a function of the elapsed time after the temperature reached at 900 $^{\circ}\text{C}.$



Fig. 4. Spectra as a function of the elapsed time after the temperature reached at 1000 $^\circ\text{C}.$

whose intensity showed increase with the increase of the wavelength. Measuring spectra of the optical thermal radiation in the electrical furnace, it was found that the spectral shape of the base-line luminescence was similar to that of the optical thermal radiation in the electrical furnace. Thus, it is the most probable that some parts of the optical thermal radiation were penetrating into the core of the optical fiber at 1000 °C. It implies that the cladding layer was damaged by the heating at 1000 °C.

4. Surface observation

Surface examination of the fiber was carried out with an optical microscope. The image of the optical fiber after being heated at 1000 °C for 15 h is shown in Fig. 5. The inserted figure shows the image of the fiber without heat treatment. Some damages were observed on the heat-treated fiber surface, and the damaged area increased with the increase of the heating time. Fig. 6 shows the ratio of the damaged area to the total surface area of the fiber as a function of the heating time at 1000 °C. The ratio increased nearly linearly with the heating time, whose behavior is similar to that of the intensity increase of the base line luminescence.



Fig. 5. Optical photographs of surface of the optical fiber heated at 1000 $^{\circ}$ C for 15 h. Inserted figure shows the image without heat treatment.



Fig. 6. Heating time dependence of ratio of the damaged area to the total surface area of the fiber.

Electron diffraction (ED) examination on the damaged surface region was carried out with the transmission electron microscope (TEM). When the heating time was less than several hours, the damaged region showed mainly an amorphous or glassy structure. The ED pattern suggested that some microcrystalline structure grew after several hours heating and the crystalline structure could be identified more clearly with the increase of the heating time. It was reported that impurities will promote crystallization of fused silica and the fluorine is typically the case as offering a nucleation site for crystallization in the glass matrix [8,9]. In the present optical fiber, the clad layer contains substantial amount of fluorine (F), which would caused the observed crystallization at 1000 °C.

Heating tests of a F doped fiber and the pure fiber was carried out in a separate experiment. The results clearly showed that the F doped fiber showed crystallization at lower temperature and with shorter heating time than the pure optical fiber. Also, the control test, namely the same experiment without exposing the fibers to the gamma-ray irradiation, was carried out. The results did not show any radiation effects, gamma-ray-enhanced increase of the base-line luminescence observed in other experiments [4,7]. This indicates that the present gamma-ray irradiation is weak and there were no explicit irradiation effects on the observed results.

5. Conclusion

The PARCO fiber had a characteristic thermal luminescent peak at 1390 nm, attributed to the OH in the silica fiber; further, the peak intensity exhibited clear temperature dependence of the Arrhenius type. The thermal luminescent spectral shape at 900 °C remained constant. Therefore, the PARCO fiber could be a candidate for a temperature measurement device up to 900 °C. Explicit irradiation effects of 5.8 mGy/s gamma-ray were not observed. The previous experiment in the JMTR [3] did not show clear evidence of the crystallization of the pure fused silica optical fiber up to 850 C for more than a month. The neutron flux and the gamma-ray dose rate were being comparable to those of ITER irradiation environment near the plasma. Thus, the PARCO fiber could be applied to the temperature measurements in ITER, where the electromagnetic noises will disturb electrical signals from the conventional thermocouples.

When the temperature was raised up to 1000 °C, the base-line luminescence in the wavelength range above 800 nm was generated and grew, suggesting some leakage of the optical thermal radiation in the electric furnace. Some damages were found on the fiber surface, which would be due to crystallization of the F doped cladding layer. The TEM examination showed that the glassy structure of the fused silica was partly crystallized after the heat treatment at 1000 °C. The crystallization of the glassy silica will be a determinant factor for the application of the optical fibers at elevated temperatures.

References

 T. Shikama, K. Yasuda, S. Yamamoto, C. Kinoshita, S.J. Zinkle, E.R. Hodgson, J. Nucl. Mater. 271&272 (1999) 560.

- [2] R.R. Dils, J. Appl. Phys. 54 (1983) 1198.
- [3] F. Jensen, T. Kakuta, T. Shikama, T. Sagawa, M. Narui, M. Nakazawa, Fus. Eng. Des. 42 (1998) 449.
 [4] A. Honda, K. Toh, S. Nagata, B. Tsuchiya, T. Shikama, J. Nucl. Mater. 367–370
- (2007) 1117. [5] D.L. Griscom, M.E. Gingerich, E.J. Friebele, Phys. Rev. Lett. 71 (1993)
- 1019.[6] O. Humbach, H. Fabian, U. Grzesik, U. Haken, W. Heitmann, J. Non-Cryst. Solids 203 (1996) 19.
- [7] T. Shikama, K. Toh, A. Honda, S. Nagata, B. Tsuchiya, SPIE Int. Soc. Opt. Eng. 6619 (2007) 66190K.
- [8] A.H. Rose, J. Lightwave Technol. 15 (1997) 808.
- [9] A.H. Rose, T.J. Bruno, J. Non-Cryst. Solids 231 (1998) 280.