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# Ion-implantation-induced densification in silica-based glass for fabrication of optical fiber gratings

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Ion implantation induces a refractive index increase in silica-based glass, which is mainly due to densification of the glass. The refractive index increase can be used to fabricate optical fiber gratings that are formed with periodic refractive index modulation in the core of an optical fiber. In this article, the generation mechanism of the densification has been investigated through measurements of thickness changes of silica glass induced by proton and He<sup>2+</sup> ion implantation. Furthermore, fabrication of the optical fiber grating using the refractive index increase has been demonstrated. From the result, ideal implantation conditions to fabricate the gratings are discussed. © 2000 American Institute of Physics. [S0021-8979(00)03922-0]

## I. INTRODUCTION

Optical fiber gratings formed with periodic refractive index changes in the core of an optical fiber have been used as various optical devices in optical fiber communication networks.<sup>1-3</sup> The optical fiber gratings can be classified into Bragg gratings<sup>1</sup> and long-period gratings,<sup>2,3</sup> depending on their grating periodicity ( $\Lambda$ ). Bragg gratings have  $\Lambda$  of less than 1  $\mu\text{m}$  and reflect light whose wavelength satisfies the Bragg condition. Bragg gratings have been applied to wavelength selective mirrors, optical filters, dispersion eliminators, etc.<sup>4-8</sup> Long-period gratings have  $\Lambda$  of hundreds of microns and couple two copropagating fiber modes, which are applied to mode converters.<sup>9</sup> Recently, coupling between the guided fundamental mode to forward propagating cladding modes by long-period gratings has been attracting much attention as wavelength selective optical filters.<sup>2</sup> The gratings are also applicable for gain-flattening filters of Er-doped fiber amplifiers.<sup>10</sup> In addition to such applications, these gratings can be used as sensors, since the length of the gratings is changed with temperature or strain, which can be detected by monitoring the wavelength of reflected or transmitted light.<sup>11,12</sup>

Currently, the optical fiber gratings are formed by refractive index changes induced by UV photon irradiation.<sup>1-3</sup> Germanium-doped silica core fibers have been used for the material to fabricate the gratings, since the fibers have relatively high photosensitivity among the currently used optical fibers.<sup>13-16</sup> The maximum refractive index change induced in a typical Ge-doped silica glass is from  $1 \times 10^{-5}$  to  $1 \times 10^{-4}$ . This value is quite high, compared with other optical fiber materials. However, a refractive index change of

$\sim 1 \times 10^{-3}$  is required for fabricating high-performance devices. Therefore, photosensitization techniques, such as H<sub>2</sub> loading,<sup>17,18</sup> have been developed. Photosensitive optical fibers, e.g., high Ge content optical fibers,<sup>19</sup> have been also used to fabricate the gratings. These facts indicate that the UV irradiation method is applicable only for specially photosensitized optical fibers.

It is well known that a refractive index increase is induced by ion implantation in silica-based glasses.<sup>20-24</sup> The maximum index increase by ion implantation is of the order of  $10^{-2}$ , which is much larger than that induced by UV photon irradiation.<sup>25-28</sup> Therefore, ion implantation can be an effective method to fabricate optical fiber gratings in all kinds of silica-based optical fibers. To establish fabrication conditions of high performance gratings, the generation mechanism of the refractive index increase should be clarified. Since the refractive index increase is mainly due to densification of the glass,<sup>23,25-27</sup> we investigate the generation mechanism of the densification in this article. Furthermore, fabrication of a long-period grating by use of the refractive index increase is demonstrated.

## II. EXPERIMENTAL PROCEDURE

The planar samples used for observation of the densification were commercially available high purity silica substrates, Suprasil-2 (Heraeus Amersil; OH content  $\sim 1000$  ppm) and ED-C (Nippon Silica Glass; OH content  $< 1$  ppm). The thickness of these samples was  $\sim 1$  mm. A Corning SMF-28 single mode optical fiber was used for the fabrication of optical fiber gratings. The core of the fiber was Ge-doped silica glass of 97SiO<sub>2</sub>:3GeO<sub>2</sub> and had a diameter of 9  $\mu\text{m}$ . The core was embedded in a cladding of pure silica glass with a diameter of 125  $\mu\text{m}$ .

These samples were implanted with protons and He<sup>2+</sup> ions. The 6 MV and the 1.7 MV tandem accelerators, both at

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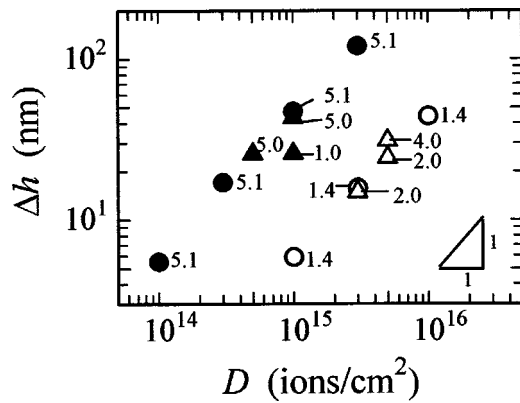


FIG. 1. Dose ( $D$ ) dependence of  $\Delta h$  observed in Suprasil-2 irradiated with protons ( $\circ$ ), Suprasil-2 irradiated with He ions ( $\bullet$ ), ED-C irradiated with protons ( $\triangle$ ), and ED-C irradiated with He ions ( $\blacktriangle$ ). Numbers next to the symbols indicate the acceleration energy (MeV) of the ions.

the University of Montreal, were used for the proton and the He ion implantation, respectively. Since the optical fiber gratings are formed with refractive index changes in the core of an optical fiber, implanted ions should reach the core. Typical optical fibers have claddings with a thickness of  $\sim 60 \mu\text{m}$ . Therefore, an acceleration energy of 2–3 MeV is required for protons and  $\sim 10$  MeV for He ions. Thus, we used acceleration energies of the order of MeV in the present experiment. The ion implantation was performed in a vacuum of  $10^{-4}$  Pa at room temperature. The beam currents of the protons and the He ions were 150 and 100 nA/cm<sup>2</sup>, respectively. Beam heating from these ion fluxes was observed. However, the sample temperature measured was always lower than 60 °C and thus the annealing effect by the ion implantation is negligible.

Ion implantation induces a thickness change in silica glass, which is due to the densification. Hence, steps are observed between the implanted and the nonimplanted regions. To measure the thickness change, we implanted ions into the sample partly covered with a metal mask and measured the step height ( $\Delta h$ ) with a DEKTAK 3030 ST profilometer, where the experimental error was  $\pm 10\%$ . The refractive index of the sample surface was measured by an Abbe refractometer at 589 nm with an experimental error of  $\pm 0.0002$ .

### III. RESULTS

Figure 1 shows the dose ( $D$ ) dependence of  $\Delta h$  induced by the implantation with protons (open symbols) and He ions (closed symbols). The acceleration energy is indicated in Fig. 1. From the results of the 1.4 MeV proton and the 5.1 MeV He-ion implantation into Suprasil-2, it was found that  $\Delta h$  is proportional to  $D$ . As observed at the dose of  $1 \times 10^{15} \text{ cm}^{-2}$ ,  $\Delta h$  observed on Suprasil-2 implanted with 5.1 MeV He ions is quite similar to  $\Delta h$  observed on ED-C implanted with 5.0 MeV He ions, indicating that  $\Delta h$  scarcely depends on the sample. The  $\Delta h$  induced by 1.0 MeV He ions was much higher than that induced by 1.4 MeV protons as

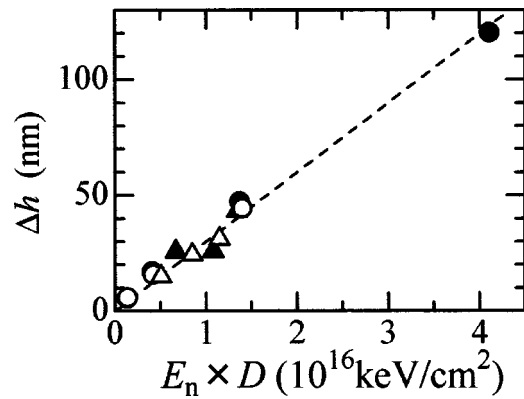


FIG. 2. Relation between  $E_n \times D$  and  $\Delta h$ . Symbols are the same as those in Fig. 1.

observed at the same dose. This result indicates that the acceleration energy itself is not a dominant factor of the densification.

Figure 2 shows the relation between  $\Delta h$  shown in Fig. 1 and  $E_n \times D$ , where  $E_n$  (keV/ion) is the energy deposited into the sample by one ion through the atomic collision process. The  $\Delta h$  is proportional to  $E_n \times D$ , and the proportionality constant is independent of the acceleration energy, the ion species, or the type of sample.

The refractive index of the Suprasil-2 was measured before and after the implantation of 5.0 MeV He ions to a dose of  $1 \times 10^{15} \text{ cm}^{-2}$ . The refractive index was not observed to increase at the surface of the as-implanted sample. However, when the sample thickness was mechanically thinned by up to  $\sim 25 \mu\text{m}$  after the ion implantation, a refractive index increase of 0.0035 was observed at that surface.

Figure 3 shows a photograph of the cross section of a proton-implanted optical fiber to a dose of  $2 \times 10^{15} \text{ cm}^{-2}$  observed by a conventional optical microscope. The protons were irradiated from the upper side of the picture and their acceleration energy was 2.4 MeV. The bright circle at the center is the light guided by the core of the optical fiber. The bright arc across the fiber seen at the depth of  $\sim 60 \mu\text{m}$  from

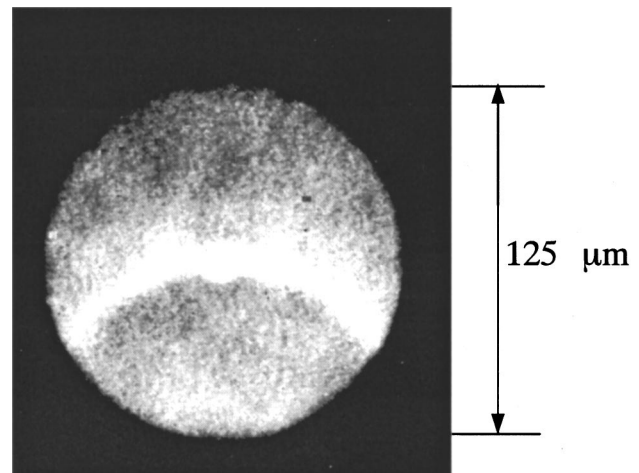


FIG. 3. Photograph of the cross section of an optical fiber implanted with  $2 \times 10^{15} \text{ cm}^{-2}$ , 2.4 MeV protons, observed by a conventional optical microscope. Upper side was exposed to the protons.

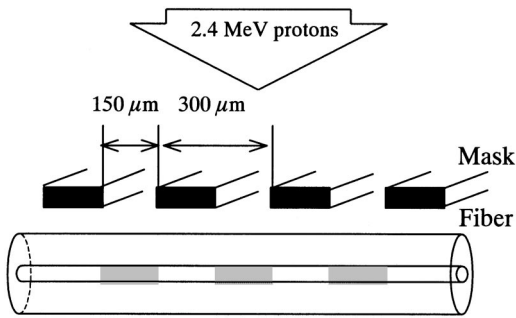


FIG. 4. Schematic of the alignment of the mask and the fiber for the fabrication of the optical fiber grating.

the surface of the optical fiber is the region where the implanted protons induced a significant refractive index increase. Here, the depth of  $\sim 60 \mu\text{m}$  coincides well with the projected range of the 2.4 MeV protons.

Fabrication of a long-period optical fiber grating was performed by use of the refractive index increase with 2.4 MeV proton implantation to a dose of  $2 \times 10^{15} \text{ cm}^{-2}$ . Figure 4 shows a schematic of the alignment of the fabrication system. The optical fiber was implanted by protons through a metal amplitude mask that had a 66 period grating with a  $300 \mu\text{m}$  pitch and a  $150 \mu\text{m}$  spacing. The transmission loss spectrum of the fabricated long-period grating is shown in Fig. 5. A very sharp loss peak at 1300 nm is observed, indicating that the fabricated grating worked as a wavelength selective optical filter.

#### IV. DISCUSSION

Implanted ions lose their energy through two major energy deposition processes, electronic excitation and atomic collision. As shown in Fig. 2,  $\Delta h$  is proportional to  $E_n \times D$ , and the proportionality constant is independent of the implanted ion and the acceleration energy. This means that  $E_n$  and  $D$  govern the densification, i.e., the densification is induced by atomic collisions.

In the case of MeV ion implantation, the energy deposited by the electronic excitation process is much larger than the energy deposited by the atomic collision process. Hence,

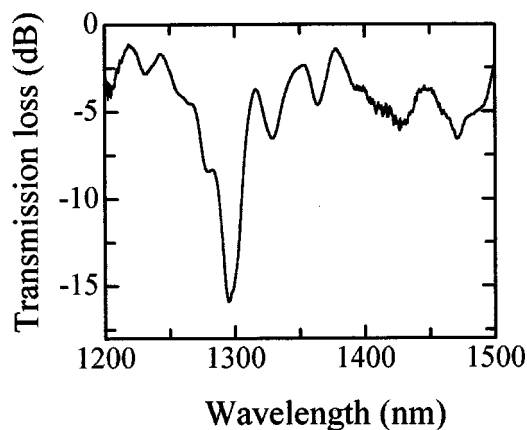


FIG. 5. Transmission loss spectrum of the long-period optical fiber grating fabricated by proton implantation.

TABLE I. Acceleration energy for several ion species required to obtain  $60 \mu\text{m}$  projected range in silica glass.  $E_n$ 's and lateral straggling of the ion species under the acceleration energy are also summarized.

Ion	Acceleration energy (MeV)	$E_n$ (keV/ion)	Lateral straggling ( $\mu\text{m}$ )
Proton	2.4	1.8	2.6
He	9.4	$1.6 \times 10$	1.4
N	$6.1 \times 10$	$1.3 \times 10^2$	$8.3 \times 10^{-1}$
O	$7.4 \times 10$	$1.6 \times 10^2$	$8.0 \times 10^{-1}$
Si	$1.6 \times 10^2$	$5.1 \times 10^2$	$7.5 \times 10^{-1}$
Ge	$4.6 \times 10^2$	$2.6 \times 10^3$	$7.4 \times 10^{-1}$

the electronic excitation process could affect the densification. However, the densification observed in the present experiment is clearly explained by the atomic collision model as shown in Fig. 2. This fact indicates that the contribution of the electronic excitation process to the densification was smaller than the measurement limit. As shown in Fig. 1,  $\Delta h$  induced by 1.0 MeV He ions at a dose of  $1 \times 10^{15} \text{ cm}^{-2}$  is 6 times higher than  $\Delta h$  induced by 1.4 MeV protons at the same dose. Here, the energy into electronic excitation process by 1.0 MeV He ions is smaller than that by 1.4 MeV protons. This result also indicates that the contribution of the electronic excitation process is quite small.

The mechanism of ion-implantation induced densification has been researched with surface stress measurements, and it has been reported that densification of silica glass induced by ion implantation is mainly due to atomic collisions.<sup>21,26,29</sup> Here, we have deduced the same conclusion from the direct densification measurement, which gives a strong validity to the previous reports. It has been also reported that the densification is scarcely affected by OH content and metal impurities.<sup>21</sup> As seen in Figs. 1 and 2,  $\Delta h$  is independent of the sample, which is also comparable with the previous report.

As shown in Fig. 3, the ion-implantation induced refractive index increase occurs around the projected range of the implanted ions. Similar depth profile of the induced refractive index increase has been reported.<sup>22,23,30</sup> Since the ion-implantation induced refractive index increase is mainly due to the densification of silica glass and the densification is induced by atomic collisions as mentioned above, the depth profile of the refractive index increase should be similar to that of the energy deposited by atomic collisions. The atomic collisions occur mainly around the depth where the ions stop. The observed depth profile thus confirms the atomic collision model. Through the refractive index measurement, it was found that a refractive index increase of at least 0.0035 was induced by 5.0 MeV He ion implantation to a dose of  $1 \times 10^{15} \text{ cm}^{-2}$ , which is quite sufficient to fabricate optical fiber gratings.

Since heavier ions have a larger  $E_n$ , they induce a higher refractive index increase with a smaller dose. Therefore, heavier ions could be effective for the fabrication of the optical fiber gratings. However, much larger acceleration energy is required for the heavier ions to reach a core of an optical fiber. Table I summarizes the acceleration energy for several ion species required to obtain a projected range of  $60 \mu\text{m}$

$\mu\text{m}$  in silica glass calculated with a Monte Carlo simulation package called TRIM. The  $E_n$ 's of the ion species under the acceleration energy are also indicated in Table I.

As shown in Fig. 5, ion implantation is clearly applicable for the fabrication of long-period gratings. However, many small loss peaks and a high background loss of  $\sim 4$  dB are also seen in Fig. 5. The structures of the loss spectrum are considered to be due to the arc shape of the refractive index increase, which forces the guided fundamental mode to couple not only to the symmetric cladding modes but also to asymmetric ones. The transmission loss structure is unfavorable for the fabrication of narrow band optical filters, while it can be advantageous for the fabrication of gain flattening filters of Er-doped optical fiber amplifiers. The background loss can be reduced by erasing the refractive index increase in the cladding. A mask with a narrow spacing, through which ions are implanted only in the core of an optical fiber, might be suitable for the fabrication of narrow band optical filters.

The ion implantation method can be applied for the fabrication of Bragg gratings. However, since the grating periodicity is less than  $1 \mu\text{m}$ , lateral straggling of implanted ions should be taken into account. As indicated in Table I, the lateral straggling of 2.4 MeV protons is  $2.6 \mu\text{m}$ . Therefore, it is impossible to fabricate gratings with periods of less than  $1 \mu\text{m}$ . As shown in Table I, heavier ions show smaller lateral straggling. Therefore, heavier ions would be effective for the fabrication of Bragg gratings. Smaller acceleration energy yields smaller lateral straggling. Hence, optical fibers or planar waveguides with thinner claddings are suitable for the fabrication of Bragg gratings by ion implantation.

## V. CONCLUSION

The acceleration energy dependence and dose dependence of ion-implantation-induced thickness changes in silica glass were examined. It was found that the thickness change was proportional to the dose and to the deposited energy by the atomic collision process. From the result, it is concluded that the densification of silica glass, which causes the refractive index increase, is induced by atomic collisions. Furthermore, successful fabrication of optical fiber gratings using the index increase was demonstrated. This method makes it possible to fabricate optical fiber gratings in silica-based optical fibers that are not photosensitive.

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