SIMS Proceedings Papers

SURFACE and INTERFACE ANALYSIS

Received: 10 September 2009 Revised: 31 March 2010 Accepted: 4 April 2010 Published online in Wiley Online Library: 22 June 2010

(wileyonlinelibrary.com) DOI 10.1002/sia.3552

SIMS analysis of Rb-doped hollow-core photonic band-gap silica fiber using a CAMECA 4550 instrument

G. M. Guryanov,[a]* A. D. Slepkov,[b] A. L. Gaeta[b] and K. W. Koch[a]

A hollow-core photonic band-gap fiber exposed to Rb vapor was studied using quadrupole-based SIMS. The Rb concentration along 30-cm fibers was analyzed using a Cs^+ beam. Effective charge compensation was obtained in the fiber cross-section without using any metal coating. A low Rb detection limit of about 6×10^{15} atoms/cm^3 was obtained in the silica microstructure region of the fiber. SIMS results revealed variations in the longitudinal distribution of Rb in fibers exposed to different loading conditions. Ion imaging of the fiber cross-section using a Ga^+ beam revealed the confinement of Rb atoms to the microstructured photonic region of the fiber, as opposed to the solid glass cladding.

Keywords: SIMS; hollow-core photonic band-gap fiber; Rb quantification; FIB imaging

Introduction

Low-light-level nonlinear optics is a key technology for many advancing applications such as single photon all-optical switches, quantum computing, and effective frequency conversion. Hollow-core photonic band-gap fiber (PBGF) is a new fiber design wherein a micron-scale air-core is surrounded by a Bragg-like hexagonal lattice of glass and air.[1] In PBGF, light is guided in and confined to the hollow core area by the optical band gap created by the microstructured region. Thus, PBGF is a very promising medium for effective light–atom interaction when the core is filled with a nonlinear material. Because of their high nonlinearities, large oscillator strengths and narrow transition linewidth, alkali atoms (Na, K, Rb, Cs) are particularly well suited for low-light-level nonlinear optics. For most photonic applications utilizing alkali atoms confined in the hollow core of PBGF, the optical density of the alkali vapor is the measurable quantity of interest.[2] It is exceedingly difficult to ascertain the alkali atomic density profile by optical measurements.

In this paper, we develop and utilize an approach to study the longitudinal and radial distribution of Rb in silica PBGF filled with Rb using a quadrupole-based SIMS tool. We use a Cs^+ beam for high-sensitivity detection of the longitudinal Rb distribution. Consistent with our previous findings,[3] this approach provides superior charge compensation and the same detection limit for alkalis in glass compared with the typical use of an O_2^+ beam. Data show almost four orders of magnitude variation in the measured Rb concentration along a 30-cm long PBGF for the case when the Rb vapor does not extend down the entire fiber. Then, the spatial distribution of Rb in the fiber cross-section is imaged using a submicron Ga-ion beam, which reveals the Rb is confined to the microstructured silica region.

Experimental

The fiber we study (Crystal Fiber Air-6-800) has a hollow core of ~6 μm in diameter, an air–silica microstructure region consisting of ~50–2 μm-scale glass structures that cover an area of ~35 μm in diameter, and a solid silica cladding of diameter ~125 μm, as shown in Fig. 1a). The experimental setup for filling PBGF with Rb vapor is described elsewhere in more detail.[4] In short, a 30-cm-long piece of PBGF is mounted between two ultrahigh vacuum optical chambers and exposed to Rb vapor at one end. Over time, Rb vapor diffuses down the hollow fiber core. Over the period of a few months to a year of continuous exposure to Rb vapor, optical measurements were regularly conducted that showed very strong low-light-level nonlinear optical interaction.[5] After 4–12 months of use at Cornell University, the fiber was removed from the vacuum system, and prepared for characterization by SIMS at Corning Incorporated.

SIMS characterization of the Rb distribution in PBGF was carried out using a CAMECA 4550 depth profiler (former ATOMIKA quadrupole-based SIMS). The longitudinal Rb distribution in PBGF was acquired using a 5 keV Cs^+ with a beam spot diameter of ~12–14 μm with a current of 10 nA. The radial Rb distribution in the fiber cross-sections was analyzed using a 25 keV Ga^+ FIB. The diameter of the Ga^+ beam was ~0.2 μm at a beam current of 1 nA. The beam sizes were measured by scanning an ion beam over a sharp edge. The beam diameter was determined from the difference in scan positions when the signal levels changed from 84 to 16% of their maximum value. For both primary beams, positive secondary ions were analyzed. The Cs^+ beam was used at a 45° incidence angle, while the Ga^+ beam bombarded the sample at normal incidence. Effective charge compensation was achieved in all measurements by using a 300 eV electron beam. The electron gun had a fixed tilt and bombarded the sample at 60°

* *Correspondence to: G. M. Guryanov, Corning Incorporated, Corning, NY 14831, USA. E-mail: guryanovgm@corning.com*

a *Corning Incorporated, Corning, NY 14831, USA*

b *School of Applied and Engineering Physics, Cornell University, Ithaca, NY 14853, USA*

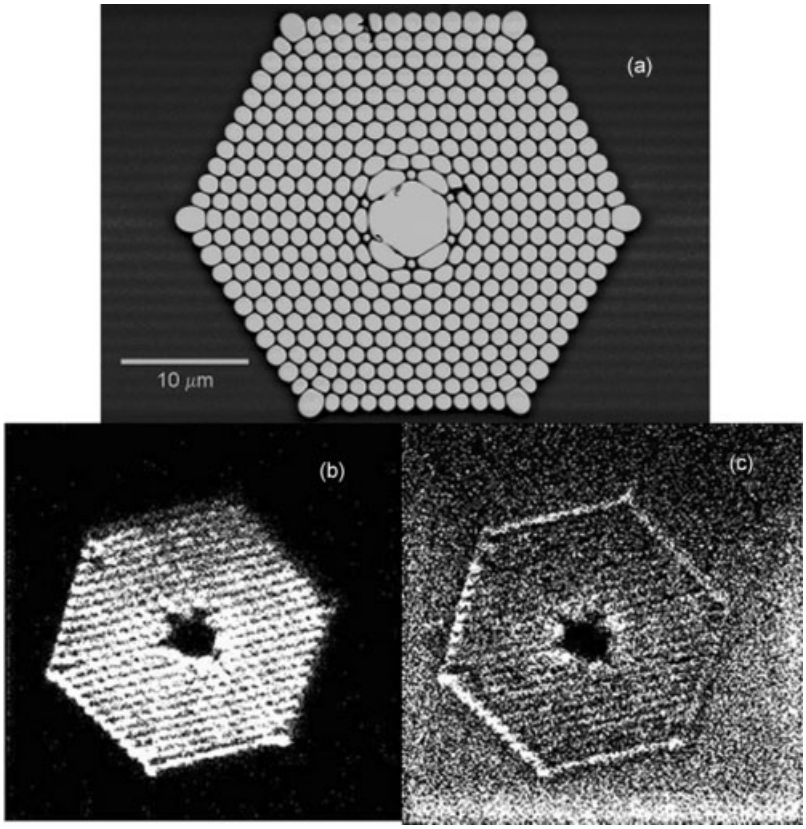

Figure 1. (a) SEM image of fiber microstructure. The black regions are glass, while the gray regions are air. The solid silica cladding surrounding the microstructure (black background) extends well beyond the displayed image crop. (b and c) 50 μm × 50 μm SIMS ion images of Rb+ (b) and Si+ (c) acquired in PBGF1 at a cross-cut depth of 5 mm from the front fiber face using 25 keV Ga+FIB. Note the absence of Rb signal outside the microstructured region.

from normal incidence. In all measurements, the ion beam and the electron beam were aligned to strike the surface at the same point. No conductive film coating was used to improve charge compensation. For quantification of the Rb analytical signal, a silica sample implanted with Rb ions was used.

For SIMS analysis, various 3- to 5-mm fiber samples were cleaved from the 30-cm-long fiber, pressed in indium foil, and mounted in a specially designed copper vice. The vice, in turn, was loaded in a standard Two-Box sample holder provided by the instrument vendor.

Results and Discussion

An example of the Rb depth profile in the ion-implanted silica reference sample using the same conditions as those for the fiber study is presented in the inset to Fig. 2. Data show that no Rb migration occurs in these conditions, and a reasonably good detection limit can be achieved in spite of using a small beam current and spot size as ultimately dictated by the small features in the fiber sample. The Rb detection limit is determined from the steady-state signal of Rb in the ^{85}Rb-ion implanted reference sample at a depth where the ion-implanted species is no longer present. Both Rb isotopes (85 and 87 amu) overlap with silicon molecular ions Si_3. The minimum ^{85}Rb detection level is limited by the contribution from Si molecular ions to 6×10^{15} at/cm^3. We use ^{85}Rb instead of ^{87}Rb in the measurements of the fiber, since ^{85}Rb has a better signal-to-noise level at low concentrations of Rb. Ion

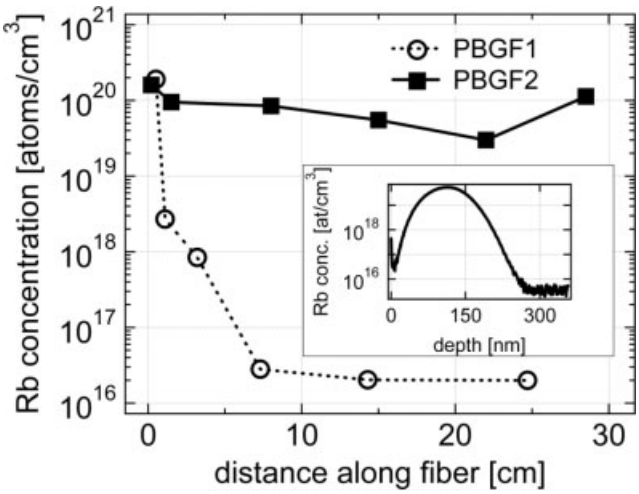

Figure 2. Longitudinal rubidium distribution in microstructured area of PBGF acquired using 5 keV Cs+beam. Inset: Rb+ depth profile for ion-implanted silica matrix calibration sample.

imaging of the fiber using the Cs^+ beam indicates that size of the Cs^+ beam is small enough to resolve the 35-μm microstructure region of the fiber from the cladding and that the Rb concentration mostly occurs in the microstructured region.

Rubidium longitudinal distributions in two PBGFs exposed to different Rb loading conditions are shown in Fig. 2. The data

show the same Rb concentration at the fiber end exposed to Rb vapor and significantly different longitudinal Rb profiles. But even the fiber with the rather fast drop of Rb concentration from one end (PBGF 1), the Rb level at the other end of the fiber is higher than the Rb detection limit obtained in these experiments (6×10^{15} at/cm^3). The second fiber (PBGF 2) shows high Rb concentration along the entire fiber length. The main difference between these two fibers (presented in Fig. 2) is the amount of time the fibers were exposed to Rb vapor and their exposure to a strong desorbing optical beam. Rubidium vapor strongly adsorbs to glass surfaces.[6] To achieve high vapor densities over extended periods of exposure to Rb vapor, it was found that a strong (~20–40 mW) 810-nm pulsed optical beam applied for several seconds efficiently desorbed Rb atoms from the glass surface.[2,4] In addition to this strong desorption beam, a lower power beam could also achieve temporary high atomic densities, but was found to be inferior compared to repeated exposure of the stronger beam. These auxiliary optical beams were not present during the measurements of the nonlinear optical interactions.

PBGF1 was in use for approximately 12 months. The strong desorbing optical beam was only applied during a short period of time at the end of the exposure to Rb vapor. PBGF2 was in use for only 6 months, but repeatedly exposed to the strong desorbing beam over the entire time. We believe the desorbing action of the auxiliary beam accelerates the diffusion of Rb down the length of the fiber. The measurement of higher Rb concentrations near the back face compared to that at the center location can be reasonably explained by the exposure of the back face to a weak Rb vapor. In our system, the front cell and back cell share an ion pump and vacuum apparatus, thus it is possible that over the course of many months, a weak Rb vapor is established in the back cell, from which some Rb may load into the fiber.

A Ga^+ focused-ion beam (FIB) is used for Rb mapping of the fiber cross-section with submicron spatial resolution. The example of such mapping using Rb^+ and Si^+ secondary ions is presented in Fig. 1b) and c). Very well resolved hexagonal structure of the PBGF is obtained and the SIMS image is very closely matched to the SEM image in Fig. 1a). The data show that Rb is completely confined to the microstructure region and does not substantially migrate into the solid silica cladding. Reducing the scan area to 7 μm × 7 μm and aligning the scan direction with the direction of the microstructure walls allowed us to obtain detailed information about Rb distribution in microstructure region of the PBGF (not shown). Strong permeation of Rb into the thin microstructured fiber walls is found. However, the beam size was not small enough to totally resolve the fine hexagonal microstructure SiO_2.

Thus, the data presented show that use of the strong optical desorbing beam is an efficient way to control the amount of Rb and its penetration down the optical fiber. Ultimately, however, the SIMS analysis only provides a proximal measure of the concentration of Rb atoms along the length of the fiber. The SIMS Rb quantification procedure is accurate only for the Rb absorbed in the silica. The Rb quantification is not accurate for Rb adsorbed on walls of the silica microstructure. Both adsorbed and absorbed species of Rb contribute to the signal level measured by the SIMS apparatus. The relative contributions from these two species cannot be determined using the current instrumentation approach. Additionally, the adsorbed Rb could be lost after removal of the fiber from the experimental set up and subsequent sample preparation described above.

The fact that Rb does not diffuse outwards from the microstructured region into the silica cladding indicates that the diffusion length scale is ~1 μm. Thus, the diffusion of the Rb down the length of the fiber must be from Rb in vapor phase, which is enhanced by the use of the strong optical desorption beam.

Conclusion

The results show that SIMS is an effective technique for analysis of Rb longitudinal and radial distribution in hollow-core photonic band-gap fiber previously exposed to Rb vapor. By quantification of SIMS data using an ion-implanted reference sample, the Rb distribution along two 30-cm fibers was obtained. Data indicated it is possible to control and measure the level and distribution of the Rb in hollow-core PBGF. A few orders of magnitude of Rb concentration variations in microstructure region of PBGF were obtained when Rb does not diffuse over total fiber length. Imaging of the fiber cross-section with Ga^+ beam revealed that the Rb totally permeated the microstructure region and there was minimal Rb diffusion into the solid silica cladding. The current study further confirmed the effectiveness of using a Cs^+ beam for alkali analysis in oxide glass matrices.

References

[1] R. F. Cregan, B. J. Mangan, J. C. Knight, T. A. Birks, P. St. J. Russell, P. J. Roberts, D. C. Allan, *Science* **1999**, *285*, 1537.
[2] A. D. Slepkov, A. R. Bhagwat, V. Venkataraman, P. Londero, A. L. Gaeta *Optics Express* **2008**, *16*, 18976.
[3] G. Guryanov, A. Pivovarov, N. Loibl, C. Schnuerer, T. Budri, U. Ehrke in, Proceeding of 20th Annual Workshop on SIMS, Key Largo, FL, **2007**, pp. 69.
[4] A. R. Bhagwat, A. D. Slepkov, V. Venkataraman, P. Londero A. L. Gaeta, *Phys. Rev.* **2009**, *A79*, 063809.
[5] P. Londero, V. Venkataraman, A. R. Bhagwat, A. D. Slepkov, A. L. Gaeta, *Phys. Rev. Lett.* **2009**, *103*, 043602.
[6] E. B. Alexandrov, M. V. Balabas, D. Budker, D. English, D. F. Kimball, C.-H. Li, V. V. Yashchuk, *Phys. Rev.* **2002**, *A66*, 042903.

