Single crystal semiconductor-core optical fiber*

Wei Wu[a], Seunghan Song[a,b], Mustafa H. Balci[a], Fredrik Laurell[c] Patrick R. Cantwell[d], John Ballato[e] and Ursula Gibson*[a, b, c]

[a]Department of Physics, Norwegian University of Science & Technology, Høgskoleringen 5, 7491Trondheim, Norway
[b]PoreLab, Department of Physics, Norwegian University of Science and Technology, Trondheim, Norway
[c]Department of Applied Physics, Royal Institute of Technology, Roslagstullsbacken 21, 106 91 Stockholm, Sweden
[d]Department of Mechanical Engineering, Rose-Hulman Institute of Technology, 5500 Wabash Avenue Terre Haute, IN 47803 USA
[e] Center for Optical Materials Science and Engineering Technologies (COMSET) School of Materials Science and Engineering, Clemson University, Clemson, SC 29634 USA

Abstract

Semiconductor-core fibers are improved by removal of grain boundaries. We use a CO_2 laser to process silicon-based cores; an alloying element reduces both the temperature and speed required for formation of a single crystal.

Keywords: semiconductor-core, laser-treated, single crystal, silicon, alloy

I. Introduction

Optical and potential THz applications of semiconductor-core fibers rely on low transmission losses, to which grain boundaries are significant contributors, as demonstrated by Healy, et al[1] and Coucheron, et al[2]. Grain boundaries act as sinks for impurities (including deposits of the interface modifiers that reduce core oxidation), but can be eliminated by laser or other thermal treatment[3] after fiber drawing. With elemental cores, a multiplicity of nucleation sites promote the formation of polycrystalline cores if there are local temperature variations and recrystallization of elemental core glass fibers is sensitive to a temperature gradient at the solidification front[4]. For smaller fiber cores a temperature gradient in the radial direction can be negligible, but because we heat the semiconductor core by conduction from the glass cladding, at larger diameters or faster scanning speed the temperature gradient in the radial direction can become significant. Especially for larger core materials such as those suitable for Thz applications, the thermal mass sets an upper limit on the translation rate at which a melt zone can be maintained. Making fibers small does not necessarily solve this problem, as it reduces the cross-section available for laser radiation absorption; extra glass is often added for studies of small cores. Fiber translation velocities of mm/s were required to establish single crystals for 12 μm Si core fiber, and that speed was insufficient to crystallize larger pure silicon cores. However, with an alloying element, small variations in the local temperature can be accommodated by changes in the local composition, which can be thermodynamically favorable to nucleating a new crystal. Thus alloys can be used to reduce the required melt zone velocity needed for single crystal growth. However, with an alloy system, the composition variation must be limited to avoid undue scattering, which can set an upper limit on the translation speed during recrystallization.

With a steep temperature gradient and a constant cooling rate (fixed power of the laser and constant velocity of the fiber), the effects of constitutional undercooling can be minimized in a solid solution such as SiGe[2]. The effects of capillarity, thermal gradient and concentration effects have been modelled by Tiller[5] and Mullins and Sekerka[6], giving:

$$v_c = \frac{D\nabla\Theta_L k}{\nabla T_L x(k-1)} \qquad (1)$$

Sixth International Workshop on Specialty Optical Fibers and Their Applications (WSOF 2019), edited by Liang Dong, John M. Ballato, Proc. of SPIE Vol. 11206, 1120615 · © 2019 SPIE
CCC code: 0277-786X/19/$21 · doi: 10.1117/12.2548428

where v_c is the critical velocity, D is the diffusion coefficient in the liquid, k is the segregation coefficient, $\nabla\Theta_L$ is the temperature gradient in the liquid, ∇T_L is the slope of the liquidus, and x is the Ge composition in the liquid. With an alloy, speeds of hundreds of microns per second permit the formation of single crystal cores with diameters up to 100 microns, of interest for solar energy and THz transmission. In this paper, we discuss the use of alloying components for the crystallization of silicon cores using solid solutions of Si and Ge.

II. Experimental

We present results on fibers with cores that are pure Si (single crystal as drawn) and with Ge concentrations of 6 at% and 0.8 at% in silicon, used to promote crystallization of the core. Fibers were drawn at Clemson University, as described in Healy's work[1]. Fibers were translated at fixed velocities (144 µm/s for 0.8%) using an LCS004 DTI piezoelectric motor with optical encoder, through the path of a 25 W Synrad CO_2 laser operated at power levels chosen to create a melt zone of width 900 µm, and an intensity chosen to avoid damage to the cladding during the anneal. Additional experiments were performed using a scanning laser system suitable for treating longer fibers (BRM 4060, 80W CO_2,100 µm/s for $S_{.94}Ge_{.06}$). In the larger system, a cylindrical lens and substantial defocus was used. Optical measurements were made using butt coupling of a fiber coupled diode laser and a Watec 902H2 camera, and X-ray characterization was performed as described in a recent article[7]. The TEM sample was prepared via in-situ lift out using a Zeiss 1540XB focused ion beam-scanning electron microscope (FIB-SEM), with final thinning done at 30 kV. Scanning transmission electron microscopy (STEM) and energy-dispersive X-ray spectroscopy (EDS) mapping were carried out on an FEI Tecnai F20ST TEM/STEM operating at 200 kV.

III. Results and Discussion

Figure 1 shows a) an STEM-EDS map which reveals a Ca-rich inclusion at a grain boundary in a SiGe fiber before annealing, b) the optical transmission of a section of pure silicon-core cane that was (111) single crystal over more than 1cm, and c) the optical transmission of a 6% Ge in silicon material after laser annealing. The alloy core was opaque prior to annealing. While both samples are highly crystalline, the mode structure of the SiGe is clearly randomized by internal scattering. Oven annealing after the laser treatment shows some promise for reducing loss[8] as it can promote Ge diffusion when slight variations exist. Reduction of inhomogeneity should both reduce loss and allow simpler mode structures.

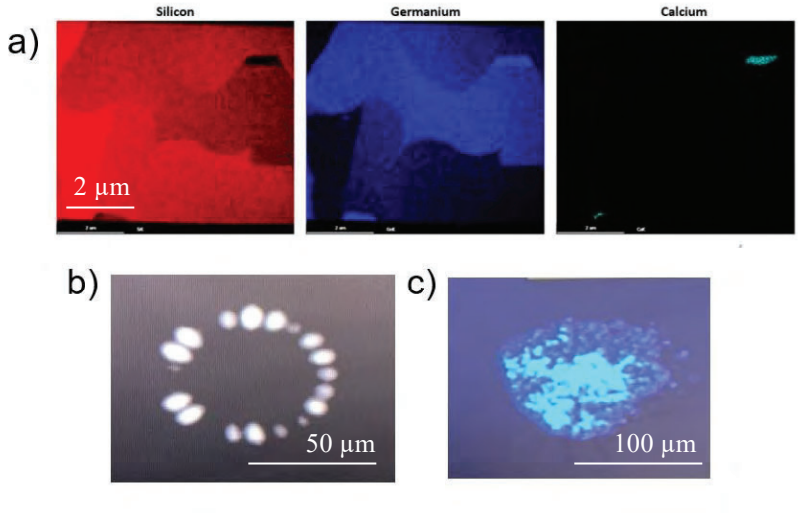

Figure 1. a) Calcium rich inclusion at an as-drawn fiber grain boundary, and optical transmission through b) single crystal silicon and c) bicrystal $Si_{.94}Ge_{.06}$.

The importance of establishing a single crystal core, to remove impurities and reduce scattering suggests using lower Ge concentration alloys. The liquidus slope approaches zero as the concentration of silicon increases, increasing the critical velocity so that the refractive index variations due to Ge concentration variation should be reduced. To test whether low concentrations would be adequate for single crystal growth, we drew fibers with 0.8 at% Ge, using commercial single crystal wafers (MTI) of this composition as the source material for the core. The cane, as drawn, was inhomogeneous, but after a single pass recrystallization, the core was seen to be single crystal and confirmed to be homogeneous using EDS mapping. XRD results for fibers are shown in Fig. 2; scans shown in a) and b) are for the fibers with optical results above. The intensity vs phi scan is an unusual presentation of the data, but gives an indication of whether a fiber has a single crystal core (and yields the axial orientation after analysis[7]). Figure 2a is a single crysta,l pure Si-core fiber, with [111] oriented along the axis (hence the 6-fold symmetry in phi), 2b is a laser annealed 0.6% Ge fiber showing a small second grain, and 2c is a scan of 0.8% Ge in Si after annealing, and is a single crystal. The brackets indicate which Bragg peak is presented for each fiber. The SiGe wafer used for these initial tests had a high conductivity, which limited IR transmission. Future work will include optimization of the preparation including higher resistivity starting materials, and/or an additional oven annealing process. In addition to solid solution forming alloys, eutectic systems may be used for recrystallizing the core, as demonstrated in reference [7].

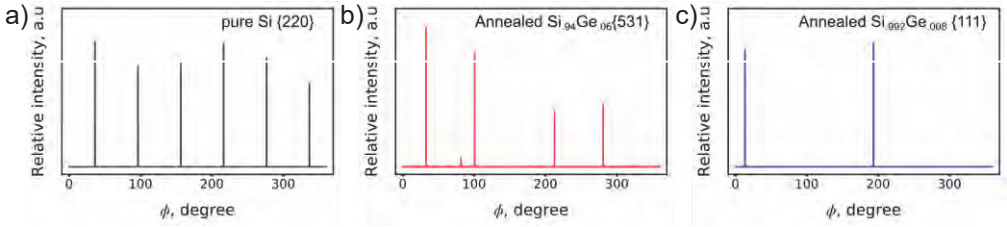

Fig. 2 XRD results on fibers a) pure silicon single crystal core fiber, b) annealed $Si_{.94}Ge_{.06}$ with small secondary crystal and c) single crystal annealed $Si_{.992}Ge_{.008}$. The {hkl} indicate the Bragg peak for each phi scan.

IV. Acknowledgements

This work was supported by Norwegian Research Council grants 219686/O70 and 262232/O70, the Norwegian Micro- and Nano-Fabrication facility, NorFab, project number 245963/F50, Swedish Research Council (grant 2016–04488), the Swedish Foundation for Strategic Research (SSF) grant RMA15–0135, and the Knut and Alice Wallenberg Foundation (KAW) grant 2016.0104. The support of the J. E. Sirrine Foundation is gratefully acknowledged. Use of the Center for Nanoscale Materials, an Office of Science user facility, was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357

References

[1] Healy, N., Fokine, M., Franz, Y., Hawkins, T., Jones, M., Ballato, J., Peacock, A. C. and Gibson, U. J., "CO_2 Laser-Induced Directional Recrystallization to Produce Single Crystal Silicon-Core Optical Fibers with Low Loss," Advanced Optical Materials **4**(7), 1004–1008 (2016).

[2] Coucheron, D. A., Fokine, M., Patil, N., Breiby, D. W., Buset, O. T., Healy, N., Peacock, A. C., Hawkins, T., Jones, M., Ballato, J. and Gibson, U. J., "Laser recrystallization and inscription of compositional microstructures in crystalline SiGe-core fibres," Nature Communications **7**, 13265 (2016).

[3] Franz, Y., Runge, A. F. J., Ren, H., Healy, N., Ignatyev, K., Jones, M., Hawkins, T., Ballato, J., Gibson, U. J. and Peacock, A. C., "Material properties of tapered crystalline silicon core fibers," Opt. Mater. Express, OME **7**(6), 2055–2061 (2017).

[4] Ji, X., Lei, S., Yu, S.-Y., Cheng, H. Y., Liu, W., Poilvert, N., Xiong, Y., Dabo, I., Mohney, S. E., Badding, J. V. and Gopalan, V., "Single-Crystal Silicon Optical Fiber by Direct Laser Crystallization," ACS Photonics **4**(1), 85–92 (2017).

[5] Tiller, W. A., Jackson, K. A., Rutter, J. W. and Chalmers, B., "The redistribution of solute atoms during the solidification of metals," Acta Metallurgica **1**(4), 428–437 (1953).

[6] Mullins, W. and Sekerka, R., "Stability of Planar Interface During Solidification of Dilute Binary Alloy," J. Appl. Phys. **35**(2), 444–451 (1964).

[7] Song, S., Lønsethagen, K., Laurell, F., Hawkins, T. W., Ballato, J., Fokine, M. and Gibson, U. J., "Laser restructuring and photoluminescence of glass-clad GaSb/Si-core optical fibres," Nature Communications **10**(1), 1790 (2019).

[8] Sørgård, T., Mühlberger, K., Wu, W., Yang, X., Hawkins, T., Ballato, J., Laurell, F., Fokine, M., Gibson, U. J. and Gibson, U. J., "Reduced loss in SiGe-core optical fibers," Conference on Lasers and Electro-Optics (2018), paper SF3I.6, SF3I.6, Optical Society of America (2018).