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The influence of core geometry on the crystallography of silicon optical fiber

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ABSTRACT

Crystalline semiconductor core optical fibers have received growing attention as greater understanding of the underlying materials science, coupled with advances in fiber processing and fabrication, have expanded the quality and portfolio of available materials. In a continued effort to better understand the nature of the crystal formation this work studies the role of the cross-sectional geometry on the resultant core crystallography with respect to the fiber axis. More specifically, a molten-core approach was used to fabricate silicon optical fibers clad in silica tubes of either circular or square inner cross-sections. In both geometric cases, the silicon core was found to possess regions of single crystallinity where specific crystal orientations persisted along a fiber length of about 4–5 mm prior to transitioning through polycrystalline regions. However, the rotation and tilting angular combination needed to align a given crystallographic axis with the fiber axis was more constant over the single crystalline region in the case of the square-core fiber while more significant variations were observed in the round-core case. This work begins to elucidate some of the microstructural features, not present in conventional glass optical fibers, that could be important for future low-loss single crystalline semiconductor optical fibers.

1. Introduction

Optical fibers possessing crystalline semiconductor cores have become a topic of recent interest internationally. Though most focus to-date has been paid to silicon [1,2], germanium [3–5], indium antimonide [6], and, more recently, zinc selenide [7,8], fibers have been realized using a variety of fabrication methods. While much work remains to further optimize their performance, interest in such fibers abounds for their consideration in non-linear optical, biomedical, and defense, sensing, and security applications [9].

In addition to this potential for technological impact, the crystalline semiconductor optical fibers offer fundamental insights into various aspects of materials science that conventional glass optical fibers do not, including, for example, solidification under highly non-equilibrium and confined conditions, the interplay between thermodynamics and kinetics, and phase equilibria to name just a few [10].

This work focuses on the role that the geometry of the core can play on the crystallinity and crystallography of crystalline semiconductor core optical fibers. Glass (i.e., silica) optical fibers generally possess cores of circular cross-section which are natural byproducts of the various chemical vapor deposition processes used in their fabrication. Additionally, cores of circular cross-section facilitate low-loss splicing and connectorization and the modal properties of cylindrical waveguides have been well-established for many years [11]. While extensive modeling of heat flow, stress and strain, and applied external electromagnetic fields has been reported in the past for fiber cores of circular cross-section, relatively little work has been devoted to fiber cores of rectangular symmetry. With the successful demonstration of optical fibers having square silicon cores, interesting device applications in nonlinear optics, image relay, or integration with planar optoelectronic circuits may be enabled.

More specifically, silicon optical fibers are drawn using silica cladding tubes that possess either a (conventional) circular cross-section or square cross-section. The underlying question posited here is whether or not the cross-sectional geometry of the core influences the crystallographic orientation of the crystalline Si core (with respect to the fiber axis). The elemental profiles, crystallinity, and crystallography of said round and square-core fibers are evaluated and discussed. Hypercircles are employed as

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a convenient and simple approach to quantifying the degree to which the square-core preform maintains its geometry during subsequent fiber fabrication. While it is found that the square-core fiber is more similar mathematically to a round fiber than a pure square cross-section, the impact on crystallinity and crystallography is discernable.

2. Experimental section

2.1. Fiber fabrication

High purity silica cladding tubes (VitroCom, Inc., Mountain Lakes, NJ) with an outer diameter of 30 mm and inner diameter of either 3.2 ± 0.2 mm (round-core) or 3.2 ± 0.2 mm flat-to-flat (square-core) were employed in this work. Silicon rods that were about 3 mm in diameter and 40 mm in length were placed into the silica cladding tubes and drawn into fiber at 1925 °C using a carbon resistance furnace purged with argon (Clemson University, Clemson, SC). The target fiber diameter was 1 mm, yielding a core size of about 100 μm, and the draw speed was 1 m/min. This molten core approach yields a fluent melt that fills and accommodates the volume and geometry of the core tube [12] hence the use of a round silicon rod inside the square-core preform is of little consequence.

2.2. Electron microscopy and compositional determination

Electron microscopy was performed using a Hitachi SU-6600 scanning electron microscope (SEM). Images were obtained under variable pressure at 20 kV and a working distance of approximately 10 mm. Elemental analysis was conducted under high vacuum, in secondary electron (SE) mode, using energy dispersive x-ray (EDX) spectroscopy in order to determine the elemental composition across the core and core/clad interface. Prior to any microscopy, the samples were polished to a 0.5 micrometer finish.

2.3. Crystallographic orientation measurement and analysis

The crystallography of the silicon core in the as-drawn fibers was measured and analyzed in an equivalent manner to that reported previously on germanium core optical fibers [13]. Briefly, samples of approximately 2 cm in length were affixed to a goniometer and first aligned vertically (within 2° of the longitudinal axis) in the x-ray beam. A Rigaku AFC8S diffractometer equipped with MoK_α radiation and a Mercury CCD area detector was used. The x-ray beam was collimated to a diameter of 0.5 mm. Upon centering, images were collected and a preliminary cubic unit cell was determined [14]. The crystallinity of the silicon core was determined using axial photographs with the fiber in its vertical orientation and, subsequently, in specific crystallographic orientations: [1 0 0], [0 1 0], [0 0 1], [1 1 0], [1 0 1], [0 1 1], [1 1 1], and [2 1 0]. The process of centering, screening, cell determination and axial orientation was repeated in 1 mm steps along the length of both the round- and square-core optical fiber.

Tilt (χ) and rotation (ϕ) angles relative to the original vertical fiber orientation required to match the above crystallographic orientations were recorded for each position along the length [13]. In order to ensure consistency in the determination of crystallographic orientation, only the angular combination with the lowest positive χ angle for each direction was determined at each consecutive position along the fiber.

2.4. Optical attenuation measurements

Transmission measurements were made at 1.3 μm in a manner equivalent to that described in Ref. 2. The output beam was imaged under 20 × magnification using an optical microscope and viewed with an IR viewer (Find-R-Scope 84499(A)-5) to ensure that the measured light was propagating in the core.

3. Results and discussion

Fig. 1 provides electron micrographs of the as-drawn round- (Fig. 1a) and square-core (Fig. 1c) silicon optical fibers. Clearly, some rounding of the edges has occurred during the drawing of the square-core optical fiber.

Since the focus of this work is to determine if non-circularity of the core can enhance the single crystallinity of (cubic) crystalline core optical fibers, then a means is needed to quantify the degree to which the core is round or square. For this purpose, Lamé curves (also known as hypercircles) are employed; see the Appendix A for more detail. Based on the measured dimensions using the electron microscope, the hypercurve exponent, n, was found to be 3 for the square-core optical fiber. While this value is mathematically closer to a circle ($n=2$) than to a true square ($n=\infty$), the core shown in Fig. 1d is more reminiscent of a square- than the round-core optical fiber of Fig. 1b; see the Appendix A for more of a comparison of this feature.

It is worth noting that fibers could be fabricated that would increase the hypercurve exponent (i.e., possess a more square-core), which could be beneficial both from the perspective of single-crystallinity and from the perspective of modal behavior. In the latter case, square-cores are somewhat analogous to round-cores in that they possess two degenerate orthogonal modes with no cut-off for the lowest order mode [15]. However, as the dimensions deviate into either rectilinear or elliptical geometries, the modal properties can be quite different [16,17] and potentially advantageous in these fibers where strong nonlinearities are expected. To the best of our knowledge, no one has studied in detail the modal behavior of a fiber as a function of such a geometric continuum; i.e., mode curves as a function of hypercircle parameter, nor in such strongly guided fibers.

The round-core silicon fiber exhibited local single-crystallinity (at individual 1 mm positions) over about 70% of the length examined whereas this was closer to 90% in the case of the square-core fiber (see Fig. 2). In both cases, the longest single crystalline length was found to be between 4–5 mm. The longest single crystal length in the square-core fiber (relative positions 3–7 mm) extended to the end of the fiber accessible for analysis so it is unclear exactly how long this grain was in its entirety. The results from the round-core fiber are consistent with those measured (not reported) on previous silica-clad crystalline silicon core optical fibers indicating that a more square cross-section promotes higher single-crystallinity.

The change in orientation between the two primary crystal grains observed in the square-core fiber appears to occur over a distinct grain boundary between the grains (at a relative position between 2–3 mm). Alternatively, in the round-core fiber there exists a longer region of polycrystallinity between 0–3 mm relative positions where many polycrystals coexist. A distinct grain boundary between two crystal grains also is observed between relative positions 7–8 mm in the round-core fiber.

There is also an observed misorientation in the longest single crystal grains of both fibers studied where the orientation gradually changes in small increments along the fibers' lengths. For the square-core fiber the maximum tilting misorientation was 1.2°, observed for the [0 0 1] and [1 1 1] directions, and the maximum rotational misorientation was 5.3°, observed for the [2 1 0] orientation. Average misorientation values for all directions were 0.9° in tilt angle and 2.8° in rotational angle. In the longest single crystal grain of the round-core fiber more extensive