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Effects of annealing on the residual stresses distribution and the structural properties of Si core fiber

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ABSTRACT

Effects of annealing on the residual stresses distribution and structural properties of Si core fiber cross-section are mainly investigated using Raman spectra map, scanning electron microscope (SEM) and X-ray diffraction. The annealing was carried out at 800 °C, 1000 °C, and 1200 °C, respectively by muffle furnace. The experimental results show that defects distribution has more obvious effects than the different characters between core and cladding on the residual stresses at the inner area of Si core cross-section. The defects distribution relate to the temperature field distribution during the process of drawing fiber. Annealing could decrease the residual stresses and make the distribution of residual stresses uniform effectively. For all samples, the Si core annealed at 1200 °C has the smallest residual stresses and the most uniform residual stresses distribution. The residual stresses of 155–437.5 MPa mainly caused by different characters between Si core and SiO_2 cladding distribute at the narrow ring area near the inner surface of cladding. This area is very likely to be composed by grains which is smaller than grains in the inner, and has looser microstructure than the inner. It exists like a buffer layer. Annealing could also improve the crystal quality of Si core. After annealed at 1200 °C, the high intensity diffraction peak of the (3 1 1) plane is generated in the X-ray diffraction spectra.

1. Introduction

Si core fiber has the potential to impact wide-ranging applications as the particularly beneficial features of crystalline silicon, such as exceptional nonlinear optics [1–5], infrared power delivery [6–11] and optoelectronic properties [12,13]. Further, it promises to be a useful platform for the incorporation of optoelectronic functionality into a new generation of all-fiber networks [14,15].

Different with the silica core fiber, the Si core and the SiO_2 cladding are different materials. The effects of SiO_2 cladding on the residual stresses and the microstructure of Si core would be mainly introduced by the boundary area of core/cladding. The residual stresses and the crystal quality are important characters of Si core fiber, because they can affect the width of bandgap [15] and the optical propagation properties [14]. Annealing is an effective way to decrease the residual stresses and improve the crystal quality of Si core. Lagonigro et al. [16] reported the Si core optical fibers annealed at different temperatures. The experimental results show that annealing leads to an improvement in the polycrystalline quality, and there is a clear reduction in the losses as the annealing temperature is increased. Gupta et al. [17] reported that a rapid photothermal processing (RPP) for polycrystalline Si core optical fibers increased the local crystallinity of the material, which improved the core uniformity and thereby causing lower losses. Healy et al. [18] reported the annealing of a amorphous Si core optical fiber by a frequency doubled Nd:YVO_4 Q-switched laser. The annealing process produces a fiber that has a highly crystalline core, whilst reducing the optical transmission losses by ~3 orders of magnitude. Another recent study by Healy et al. [19] reported the annealing of a Si core optical fiber by a CO_2 laser. This method can produce single crystal Si cores with a length of 1.8 cm with optical transmission loss of 2 dB/cm at 1.55 μm and 1 dB/cm at 2.0 μm. Ji et al. [20] reported the creation of single-crystal silicon core fibers by laser crystallizing amorphous silicon deposited inside silica capillary fibers by high-pressure chemical vapor deposition. The single crystal fibers have very low optical losses down to ~0.47–1 dB/cm at the standard telecommunication wavelength (1550 nm).

However, there are few reports about the effects of annealing on the crystal characters and residual stresses distribution on the cross-section of Si core. In the report of Gupta [17], Raman measurements were carried out on various locations on the as-drawn and RPP-treated Si core fiber which have a diameter of 1.6 mm. For the as-drawn Si core fiber, Raman shift varies with the location, suggesting the presence of

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stresses across the core and non-uniform distribution of the residual stresses. However, the observed shifts in silicon Raman frequency in the as-drawn fiber were no longer present in the RPP-treated fiber. The RPP treatment increases the local crystallinity and therefore assists in the reduction of local stress field in the core, leading to a more homogenous fiber.

In our study, the Si core fiber with a diameter of 180–200 μm was drawn by the molten core drawing (MCD) method, and then annealed in a muffle furnace. The residual stresses distribution on the cross-section of Si core and the structural properties of as-drawn fiber and fibers annealed at different temperature were mainly investigated by Raman spectra map, scanning electron microscope (SEM) and X-ray diffraction. Our work aims to better understand the effects of annealing on the residual stresses distribution, crystallization characterises of Si core, and the mechanism of residual stresses formation, in order to obtain Si core fiber with more homogeneous crystalline microstructure and less residual stresses.

2. Experiments and measurements

Fiber samples: The Si core fiber was fabricated by the MCD method. Si bulk crystal with purity of 99.99% was packed into a silica tube having an inner diameter of about 3 mm and an outer diameter of 10 mm, which had been pre-drawn in order to seal one end of the tube. The fiber was drawn using a drawing tower at approximately 2200 °C which is above the melting point of the Si core, and the molten Si was then encapsulated by the viscous silica cladding. The drawing speed is about 5 m/min (8.3 cm/s). Approximately fiber of 3 m length and 180–200 μm diameter was drawn, which yielded a core size of 25–45 μm. Fig. 1 shows a microscope photograph of the as-drawn Si core fiber.

Fiber annealing treatment conditions: The Si core fibers were annealed at 800 °C, 1000 °C, and 1200 °C, respectively in a muffle furnace. The heating rate was 10 °C/min. The fiber was kept at the target temperature for 20 min. After that, the fiber was cooled down to 400 °C at a rate of 2.5 °C/min. Then, the furnace was shut down to chill the Si core fiber naturally to room temperature.

SEM and energy dispersive X-ray spectroscopy (EDS) characterisation: The cross-section of the fiber was imaged using SEM. Electron microscopy was performed using a CamScan Apollo 300 scanning electron microscope. The electron microscope was operated at 10 mm working distance. EDS was used for elemental analysis to examine the distribution of elements across the core/clad interface. Elemental compositions were measured at several locations traversing the cross-section of the fiber, and the distribution of Si, and O elements were examined.

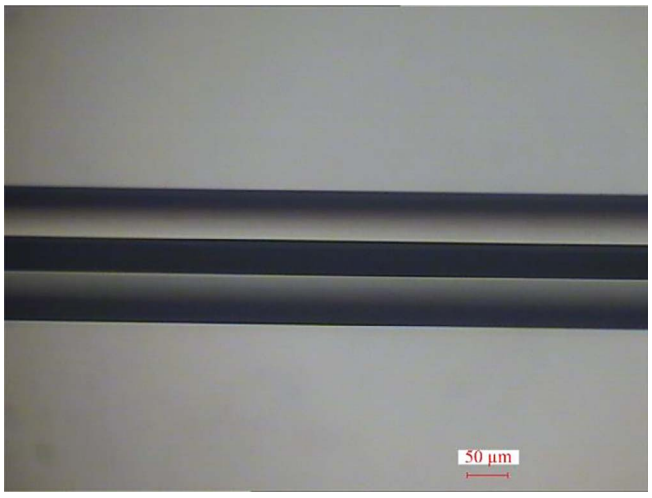

Fig. 1. The microscope photograph of the as-drawn Si core fiber.

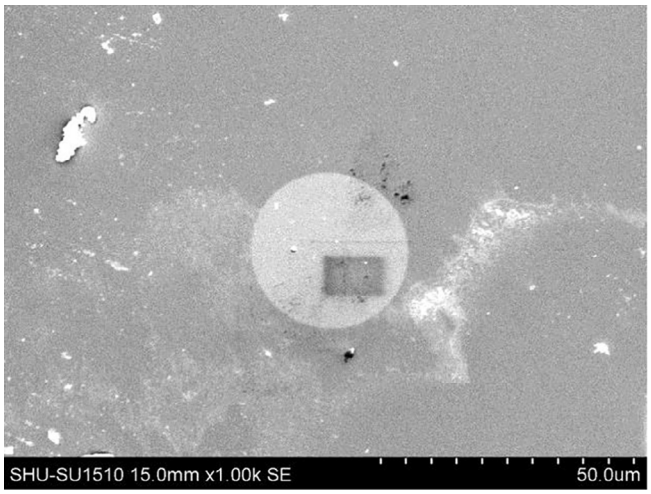

Fig. 2. The cross-section of the Si core fiber annealed at 1200 °C.

Raman spectra map analysis: Raman spectra map were collected on both the Si starting bulk and the cross-section of Si core at room temperature in the backscattering configuration using a LabRam HR800 spectrometer from Horiba Jobin Yvon equipped with a Peltier-cooled CCD detector, an 1800 lines/mm grating, a He-Ne laser with an excitation wavelength of 633 nm, an optical microscope and a motorized X-Y stage. The incident laser light is focused onto the sample's surface through a 50x objective. The spectra were fitted by a Gauss-Lorentzian function.

Micro-region X-ray diffraction analysis: micro-region X-ray diffraction analysis was used to study the crystallization state of the Si core. It was carried out on the Si core cross-section using an 18 KW D/MAX2500V + /PC X-ray diffractometer with Cu K_α radiation (λ = 1.5418 Å) and a solid-state Ge detector. Diffraction patterns were collected in 0.02° steps from 20° to 80° in 2θ.

3. Experimental results and discussion

The cross-section of the Si core fiber annealed at 1200 °C is shown in Fig. 2. The core size is about 30 μm. The core is circular and there is a strong contrast between the Si core and the silica cladding. Moreover, there are no obvious discontinuities at the core/cladding interface or obvious cracks or signs of bubbles in the core.

Elemental analysis was performed on the cross-section of the fiber annealed at 1200 °C, as shown in Fig. 3. The Si concentration of the core is almost 100% with negligible oxygen, and the interface is well-defined.

To determine the residual stresses and the crystal quality of the Si core fibers, Raman frequency map and FWHM value map were carried out on the cross-section of Si core fibers. A Si bulk crystal was measured as a reference. There are big differences between SiO_2 cladding Raman frequencies and Si core Raman frequencies. In Raman frequency maps, the different values are represented by different colors. For more obvious contrast of colors at the area of Si core, if the Raman frequencies of SiO_2 cladding is smaller than 519 cm^{-1}, they are set to 519 cm^{-1}. Similar to this, In the FWHM value maps, if the FWHM values of SiO_2 cladding are larger than 4.2 cm^{-1}, they are set to 4.2 cm^{-1}.

The Raman frequency maps and the values on the cross-section of Si core are as shown in Fig. 4 and Table. 1, respectively. All fibers and Si bulk crystal exhibit a strong peak around 520 cm^{-1}. The Raman frequency of bulk crystal is 521 cm^{-1}. For the as-drawn Si core fiber, the Raman frequencies are ring distribution, and the frequency step can be observed on the cross-section of Si core. The Raman frequencies of the area (area 1 in the image) near the middle of the cross-section are