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Fabrication of chalcogenide glass photonic crystal fibers with mechanical drilling

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ABSTRACT

A mechanical drilling method for the preparation of photonic crystal fibers (PCFs) is presented in this paper. Several PCFs were fabricated with $Ge_{20}Sb_{15}S_{65}$ chalcogenide glasses, which have high transparency in the mid-infrared (IR) range. The mechanical drilling method has been identified as a powerful tool to prepare fibers with a variety of structures and to increase the transmission of the obtained fibers. For a three-ring PCF, the near-field intensity distribution and the transmission loss were measured. It was found that most of the optical energy is contained in the core of the PCF. The profile of the near-field intensity image shows that the prepared PCF can work with a large mode area, which is important in high-power laser propagation and fiber amplifiers.

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1. Introduction

Chalcogenide glasses are known for their large transparency to light of mid-infrared frequencies, which includes the two atmospheric windows lying from 3–5 μm and 8–12 μm [1]. A remarkable property of chalcogenide glasses is their strong optical nonlinearity. Depending on composition, the nonlinear coefficient n_2 can be 100–1000 times larger than for silica glass [2,3]. Another crucial advantage of chalcogenide glasses is their ability to be drawn into optical fibers, as has been demonstrated during the last decade. Among the serious applications of these optical fibers, photonic crystal fiber (PCF) is currently particular attractive for its novel features [4–6].

PCFs, also called "microstructured" or "holey" fibers, provide a large number of unique properties such as endlessly single-mode or single-mode with adjustable mode field diameter (MFD). Large MFDs are useful to minimize the risks of glass damage during transport high power laser beams and small MFDs enable the enhancement of nonlinear effects [7]. Furthermore, the chromatic dispersion in PCFs can also be managed by modifying their geometric profiles [8]. Depending on fiber design, chalcogenide PCFs can be used for power delivery [9], broadband sources [10–12], pollutant detection or optical sensors [13].

There are several common techniques to produce PCFs. The first is the "stack and draw technique," which is widely used to make silica fibers and chalcogenide PCFs [14–16]. This technique has the ability to produce microstructured performs with a large number (>60) of air holes, but this necessitates a large amount of difficult handling. Furthermore, studies have shown that PCFs fabricated with this method possess a high optical loss, due to inhomogeneities at the interfaces between capillaries [17]. Casting method is an useful technology to avoid the inhomogeneities and has been successfully used to prepare low-loss chalcogenide microstructured fibers [18], but this method needs a very carefully manufacturing of the molds and fixes them into the ampoules. The third technique is the extrusion method [4,19], which seems simpler than the "stack and draw", but is still quite challenging when aiming for preforms with very complex microstructure patterns. Drilling technique is another powerful method to produce preforms, which has been successfully used to prepare high quality microstructured optical fibers with various materials, such as As_2S_3 chalcogenide glasses [20] and polymers [21].

In this paper, chalcogenide PCFs with different structures were fabricated with mechanical drilling method. $Ge_{20}Sb_{15}S_{65}$ chalcogenide glass was specially chosen as the background material, which is a non-arsenic environment-friendly glass material, and can still possess high transmission property, good thermal stability and good mechanical stability in infrared range. The method based on mechanical drilling allows the preparation of various PCF structures by choosing special drills. More importantly, this method can increase the transmission of the obtained PCFs, as inhomogeneities at the interfaces are greatly reduced. When the fibers in this study were drawn, the near field intensity distributions of the fibers were observed by using a microscope objective and a CCD camera. The result showed that most of the optical energy could be confined to the core of the PCF, and that the PCF could work with a large mode area.

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2. Preform and fiber fabrication

2.1. Glass synthesis

Chalcogenide glasses were prepared using the melt-quenching method in silica ampoules under vacuum [22]. High-purity raw materials were used for glass preparation, i.e. germanium, antimony, sulfur and selenium 5 N. The starting materials were distilled in different tubes, and sulfur was thoroughly purified by performing multiple distillations to eliminate water and carbon, which hindered the transmission of glasses. After purification, the different elements were sealed in the same silica ampoules under vacuum (10^{-3} bar). The ampoules were then heated in a rocking furnace at 950 °C for 10 h and then cooled to 650 °C for 1 h, allowing the fusion of the elements and the homogenization of the melt. After then, the ampoules were quenched in ice water to allow glass formation and to avoid crystallization. Finally, the vitreous sample was annealed at a temperature slightly below the glass transition temperature ($T_g - 10 = 286$ °C) for 6 h and then cooling to room temperature, the cooling speed must be very slow and this process will last more than 10 h. The glasses were then cut into 2-mm-thick disks by using a diamond wire saw (Well MURG 24-A, Switzerland), and their two parallel sides were optically polished using 1 μm polishing film for the measurement of their thermal and optical properties.

The glass transition temperature, T_g, was measured with a differential scanning calorimeter (DSC). T_g was measured to be 296 °C, as indicated by Fig. 1, which shows the thermal flux versus temperature. It can be found that, between 296 °C and 450 °C, this composition exhibits no crystallization peak in the DSC curve when heated at a rate of 10 °C/min, which validates the good thermal stability of our prepared glasses. The microhardness of the glass was another important property in the fabrication of PCF preforms by using mechanical drilling. It was measured with a charge of 25 g for 5 s by using a Vickers Microindenter (Everone MH-3, Everone Enterprise Ltd., Shanghai, China). The average hardness of the prepared glass was measured to be 210.1 kg mm^{-2}, which is softer than the traditional silica glass, so the drilling parameters should be carefully adjusted in the fabrication of preforms.

2.2. Preform fabrication

Drilling techniques have been successfully used to prepare high quality microstructured optical fibers. This method can manufacture preforms with various microstructures from simple ones to more complex ones. In this study, mechanical drilling equipment was developed for the preparation of chalcogenide glasses preforms with a variety of geometrical patterns.

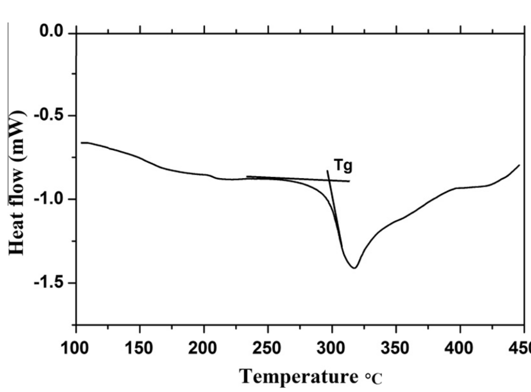

Fig. 1. Determination of transition temperature T_g by DSC measurement.

The scheme of the mechanical drilling is shown in Fig. 2. The equipment consists of two main parts: a bit for drilling and a precise *x*–*y* platform for position control of air holes. In order to prepare preforms with various patterns, twist drill bits with different diameters were used. The rotation speed of the bit is an important parameter, and should be very carefully adjusted. For the air holes with diameter of 2 mm, it is found that the rotation speed cannot exceed 1000 r/min, otherwise the glass will crack. The rotation speed can be much higher for smaller holes. In this work, the 1 mm diameter holes were prepared with the rotation speed of 1500 r/min. The forward speed of the bit was also very critical. It was found that a proper forward speed of the bit was 2 mm/min for the prepared glass in our work. A water cooling system was introduced to eliminate heat generated by friction between the glass and the broach during drilling to avoid gradual embrittlement of the glass rod. With this cooling system, the glass can keep low temperature during the drilling. The prepared chalcogenide glass has good chemical stability at normal temperature, it will not react with water during the drilling. So the prepared preforms can keep good optical transmission after drilling. The quality of the inner surface of the holes is also critical for the quality of the subsequently drawn fiber. After drilling, the inner surface of the air holes were further polished using a special broach. When the drilling is finished, the preform needs to be annealed again at a temperature slightly below the glass transition temperature for 5 h before being slowly cooled to room temperature. By optimization of the drilling parameters, especially rotation speed and forward speed, the drilling of a regular pattern of holes in a glass rod can be obtained.

Various patterned preforms were prepared in this way, such as microstructured fibers with several rings of holes in a triangular pattern (6 holes, 18 holes and 36 holes), and preforms for other microstructured fibers including holes with different hole diameters. For all these preforms the outer diameter is 18 mm and the length is about 50 mm. By choosing special bits, the diameters of the air holes can range from 0.8 mm to 2 mm, leading to an adjustable range of air hole ratio d/Λ between 0.25 and 0.6. Fig. 2(b) and (c–e) show the chalcogenide glass rod and the prepared preforms. The results of the experiment have shown that some PCF structures with closely spaced air holes can be obtained, which validates the excellent mechanical stability of our prepared glasses.

2.3. Fiber drawing

The preforms were drawn into PCF with a fiber-drawing tower (TGL-A, China). The properties of chalcogenide glasses are significantly different from those of traditional oxide glasses. Many parameters need to be carefully controlled during PCFs drawing, such as preform temperature, translation speed, fiber drawing speed, pressure in the holes, flow rate of inert gas circulating along the preform etc.

In the drawing process, a suitable temperature is the key parameter to obtain a fiber without crystallization. It is shown above that the glass transition temperature T_g is 296 °C, and no crystallization peak appeared up to 450 °C. Hence, fibers are expected to be drawn at any temperature between 296 °C and 450 °C. In general, fiber drawing should be performed on the condition that glass viscosity is between 10^4 pa s and 10^5 pa s [24]. In our work, the drawing temperature is chosen to be 435 °C at which the viscosity of the prepared glass is around 10^5 pa s. Fig. 3 shows the SEM image of the prepared PCFs that were drawn from the preforms obtained by using mechanical drilling. X-ray diffraction (XRD) of waste materials from fiber drawing revealed no crystallization peaks, which validated the good thermal stability of our prepared glasses.

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