Fabrication of lead-gallium-bismuth (PGB) optical fibre for mid-infrared nonlinearity applications

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Abstract. Lead-gallium-bismuth (PGB) is a non-silica based glass that can be used in the fabrication of optical fibre with high nonlinearity, low transmission loss, high thermal stability, and a broad transparency window. With a nonlinearity two orders higher than that of the fluoride based fibres, it has attracted much attention worldwide for its potential application in the mid-infrared region, particularly for applications such as supercontinuum generation. The fabrication process of PGB optical fibre is immature, leading to relatively high transmission loss and limiting the output average power to a few hundreds of milliwatts. However due to its intrinsic advantages such as high nonlinearity, high thermal stability, and low transmission loss, there is huge potential to achieve a higher output power by further improving the fibre fabrication processes. In this paper, we document the design and demonstration of a PGB optical fibre fabrication aimed for the delivery and nonlinear applications in the mid-infrared region. Fabrication steps from glass melting, preform making and fibre drawing will be covered in detail. By optimizing each of the fabrication steps, we expect to be able to produce PGB optical fibres suitable for supercontinuum generation and other nonlinear processes in the mid-infrared region.

1. Introduction

The transmission of optical signal of wavelength above 2 μm in conventional silica based optical fibre is limited because of the material absorption of silica is too large. On the other hand, non-silica glass fibres have the advantage of lower phonon energy in mid-infrared regions (e.g. chalcogenide-based glasses: 300–450 cm^{-1} and fluoride-based glass: 560 cm^{-1}) than the silica fibre (silica glass: 1100 cm^{-1}), thus providing a broader transparent window into mid-infrared region. Table 1 shows the refractive index, the third order nonlinear optical coefficients and nonlinear refractive index of various materials. In this chapter, we document the design and demonstration of a glass system of PGB optical fibre fabrication aimed for the delivery and nonlinear applications in the wavelength region of 2 μm and above.

Table 1. Comparison of nonlinear parameters of various materials

Material	$\lambda_{measured}$ (μm)	n_0	n_2 (m^2/W)	reference
Fused Silica	1.55	1.44	2.79×10^{-20}	[18]
Schott LLF1	1.55	1.53	6.0×10^{-20}	[21]
Schott SK2	1.24	1.59	2.1×10^{-20}	[22]
Schott F2	1.24	1.6	2.9×10^{-20}	[22]
Schott SF6	1.55	1.76	2.2×10^{-19}	[21]
Schott SF57	1.55	1.8	4.1×10^{-19}	[21]
Tellurite	1.06	2.03	5.1×10^{-19}	[17]
PGB	1.55	2.3	$\sim 10^{-18}$	[9]
GLSO	1.52	2.25	1.77×10^{-18}	[23]
GLS	1.52	2.41	2.16×10^{-18}	[23]
AsS	1.55	2.44	2.0×10^{-18}	[17] [19]
AsSe	1.55	2.83	1.1×10^{-17}	[20]

Linear and nonlinear indexes (n_0 and n_2) at measured wavelength ($\lambda_{measured}$).

Another additional advantage of non-silica glass fibres are their high nonlinearity. Glasses with large optical nonlinearities have been obtained in glass systems such as fluoride [1], and chalcogenide glasses [2]. Comparatively low loss mid-infrared transmission in fluoride based fibre is achievable, but the nonlinear refractive index of such fibre is not considerably large. As such, in applications which require high nonlinearity, a long piece of fibre is necessary. Chalogenide glasses exhibit high nonlinearity but it is difficult to find a suitable laser source close to its zero dispersion wavelength.

Heavy metal oxide glass systems with both high nonlinear refractive index and zero dispersion wavelengths close to conventional laser source such as tellurite or gallate glasses should be good candidates to achieve efficient nonlinear generation. Tellurite glass fibres were demonstrated for their high nonlinearity applications in the mid-infrared [3, 4].

Gallate glass containing lead and bismuth oxide (PGB) was reported to have the highest χ^3 of other oxide glasses. Glass systems with only lead and bismuth oxides are unstable with respect to crystallisation. Additional portion of Ga_2O_3 plays the role of the glass former, however too much percentage of it would degrade the glass refractive index. W.H. Dumbaugh completed a series of studies of PGB glass compositions [5-7] and reported good glass forming composition with high refractive index. However, PGB glass fibres have not yet been demonstrated. Ducros et al. [8, 9] demonstrated holey fibres based on PbO-Bi_2O_3-Ga_2O_3-SiO_2-CdO glass compositions. Additional SiO_2 made the composition more stable against devitrification. However, SiO_2 has strong absorption at the wavelength around 3.0 μm and moves the multi-phonon absorption edge toward the shorter wavelength. Thus in this paper, we focus in the glass forming system without any SiO_2 added.

2. Glass melting of lead-gallium-bismuth glasses

Various glass compositions each with different ratio of PbO, Ga_2O_3 and Bi_2O_3 were chosen to analyse their transmission and physical properties. In our glass melts, PbO, Ga_2O_3 and Bi_2O_3 are powders obtained from chemical suppliers with purity of 99.999%. The powders are carefully weighed separately and batched in a glove-box controlled environment. The batched samples are then mixed for at least 2 hours using a roller mixer. The mixed sample is checked visually for homogeneity and the content is loaded into a chosen crucible. In all of our glass melts, we used either alumina or platinum crucibles. The batched sample is melted at 1050^{o}C in the crucible for one hour under 3 *l*/min oxygen-nitrogen premix gas purging. During the push out from the melt furnace, the melted glass is either poured into a mold for casting or left in the crucible to quench to room temperature.

From the resultant glass obtained from our melts, we can see that glass melts from platinum crucibles have the characteristic orange appearance while glass melts in alumina crucibles have the characteristic yellowish appearance. This colour difference is caused by reactions of the glass melts at the molten temperature with the crucible material. This is the reason why we try to keep the glass melting time to a minimum.

It is very important to know the thermal properties of the glass we produced. Different from very stable silica glass systems, non-silica glass such as our PGB glass is easily crystallized. Glasses that we melted will need to go through various heat treatment processes such as extrusion, preform canning and fibre drawing. To obtain the thermal properties of our glass samples, we carry out differential thermal analysis (DTA). From the analysis, we can find out the region of glass transition, crystallization and melting points. This is useful to determine the annealing temperature for future melts and determine the temperatures of processes such as annealing, extrusion, preform canning and fibre drawing. The glass has a glass transition temperature (T_g) around 330°C and crystallization peak (T_p) presented at 500°C. When the glass is heated to the transition temperature, there is a chance that crystals will start to form in the sample. Above the transition temperature, the rate of crystal growth will increase to the crystallization peak. Thus during all these heat treatment fabrication processes, we have to ensure that the glass sample stays a minimum duration above the glass transition temperature.

To achieve the step index profile of the fibre design, we identified glass compositions within the glass formation region, which give different refractive index for the core and the cladding. We also have to take into consideration the physical properties of the chosen composition as the glass has to go through a series of heat treatment processes (casting, extrusion, canning and fibre drawing) that may cause crystallization in the glass preform of resultant fibre. For the purpose of optical fibre fabrication we have identified two PGB glass compositions for the fibre core (n=2.22) and cladding (n=2.16) structure.

3. Fibre preform fabrication

The approach used for preform fabrication is the rod-in-tube method to obtain the core-clad structure of the step indexed fibre. The molten glasses are cast into a pre-heated stainless steel casting molds. The stainless steel cast is cleaned and pre-heated. Glass melts are allowed to quench, and glass annealing is done immediately after casting.

Figure 1. shows an example of a glass billet removed from the stainless steel mold after quenching and annealing.

Figure 1 - Example of a glass billet casted for extrusion

Through the extrusion process, we can produce PGB rods and tubes of various diameters for our rod-in-tube fibre drawing method. Some examples of the resultant of tube and rod extrusions are shown below.

Figure 2 - PGB glass after rod extrusion

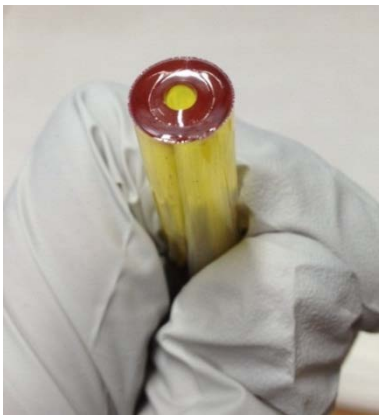

Figure 3 – PGB glass after tube extrusion

Apart from fabricating PGB glass rods and tubes for the rod-in-tube method in fibre drawing, a suspended core preform design is also implemented to produce PGB suspended core fibres.

Figure 4 – (a) Extrusion die for suspended core preform; (b) Extrusion die after extrusion run; (c) Cross-section of extruded preform

Because of the more complex design as compared to the rod and the tube, the extrusion speed of the suspended core is set to a much lower speed of 0.06 mm/min. The slow extrusion speed will help to maintain the shape and structures of the resultant preform.

4. Drawing of lead-gallium-bismuth optical fibre

The fibre preform for step index fibre is made by putting together the extruded rod and tube of the respective core and cladding glass composition. The complete preform is then drawn on the fibre drawing tower at the temperature of 530^{o}C and gas flow of O_2/N_2 during the process.

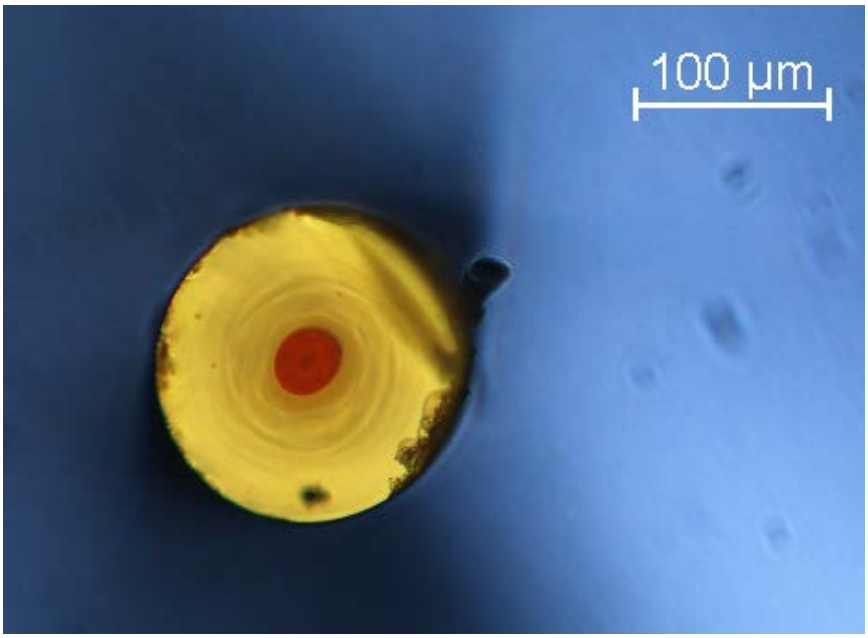

Figure 5- Cross-section of the fibre drawn

The resultant fibre of the process has a cladding diameter of around 160 µm and core diameter of around 35 µm. We noticed the shape of the core is not round, this could be because the drawing temperature is too high or vacuum applied between core and cladding is not even. To observe the light guiding in the core, we launched 1550 nm of laser light into a 50 cm length of fibre and used a CCD detector to observe guidance of light in the core.

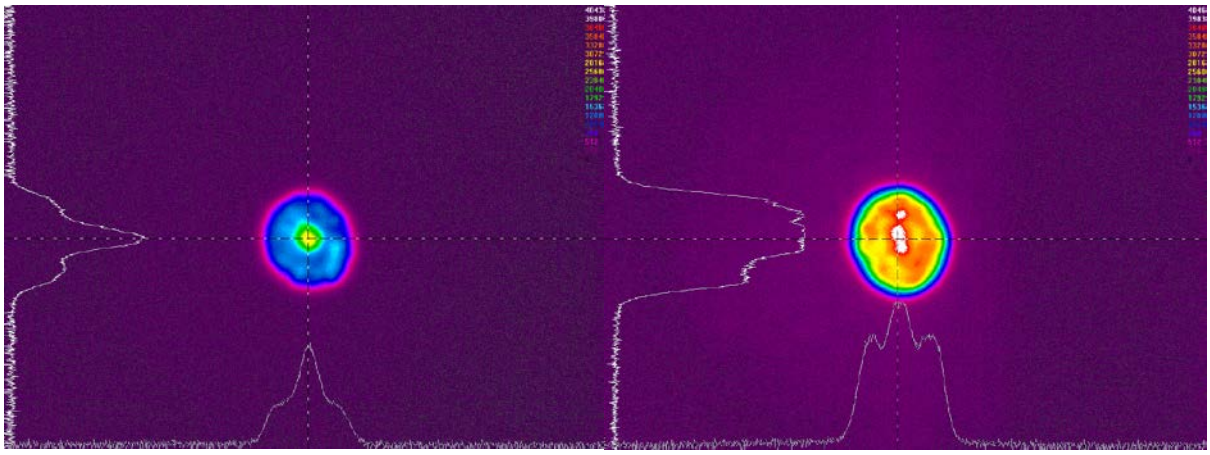

Figure 6 - CCD image of the fibre cross-section with 1550 nm laser

The step index fibre design discussed above is simple and easy for us to produce the preform using the rod-in-tube method, using two slightly different glass compositions to obtain the difference in refractive index. However, it is difficult to produce fibre that has a small core using this method.

The wagon wheel suspended core design is formed with the fibre core suspended in an air space in the fibre by only three glass arms attached and evenly spaced apart. The light in the core is guided within the air-core interface. Using this suspended core fibre design, we will be able to eliminate the necessity to have two different glass compositions in the fibre preform for fibre drawing, allowing us to use a more optimized condition for the core glass during the process. The suspended core structure preform can be fabricated by the extrusion process. This fibre design will also allow us to have a much smaller core diameter without additional heat treatment process. Careful control of the parameters during extrusion allows us to produce the fine structures of the small core preform.

The setup for fibre drawing of the suspended core PGB fibre is similar to the step indexed fibre. The extruded suspended core preform is reduced to the size of around 2 mm outer diameter by cane pulling on the fibre drawing tower. The cane is then in turn inserted into an extruded tube of PGB glass of the same composition. The preform combination is then drawn down to fibre size of 100 – 200 µm.

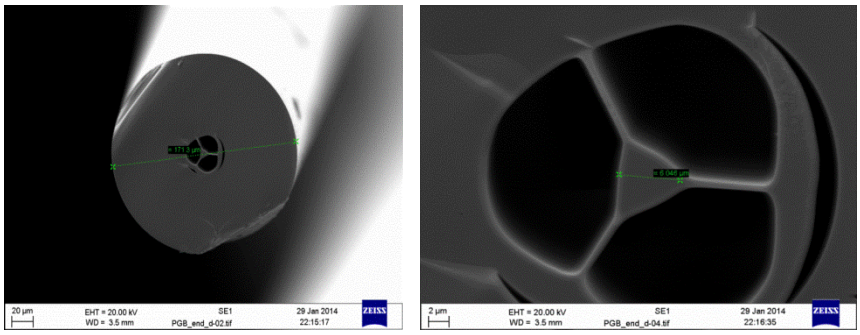

Figure 7 – Resultant fibre of PBG suspended core fibre draw

Figure 7 above shows the resultant fibre cross section. Two ends of the cane are fused before the draw such that air is being trapped inside the air holes during the whole fibre drawing process. A separate vacuum of 20 mbar is applied to the gap between the cane and the tube to collapse the gap during the fibre draw. The resultant fibre has an outer diameter of 170 µm and a core diameter of 6 µm.

To observe the light guiding in the core, we launched 1550 nm of laser light into a 50 cm length of fibre and used a CCD detector to observe guidance of light in the core.

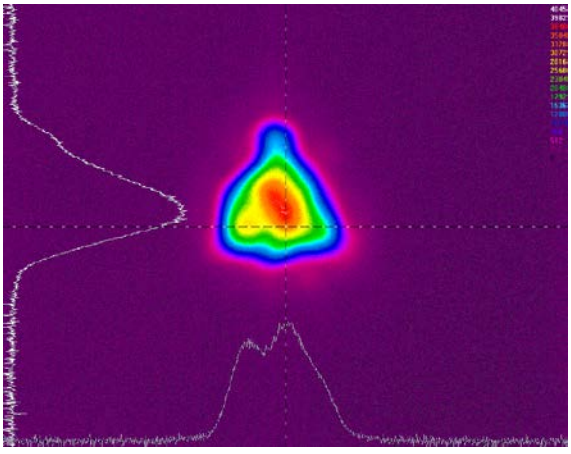

Figure 8 - CCD image of the fibre cross-section with 1550 nm laser

Conclusion

We have successfully designed and fabricated both step index and suspended core PGB glass fibres. The fabricated fibres have been tested and shown to guide light well. In the process of glass making, we looked into the glass forming region of the glass system and singled out the glass composition that suits our requirement of physical and thermal stability; and simultaneously giving us high reflective index and nonlinearity. Various parameters of the glass melting conditions were also tested to allow us to successfully produce quality glass bulks for the fabrication of fibre preforms with varying refractive index. We performed various processes on the glass melts to form the fibre preform we require. Among them, the extrusion process allows us to shape glass melts into the shape of rods, tubes and even microstructure designs. Casting is another method we used for preform fabrications. With the various parts of the preform made to shape, we are able to attach them together forming the core-clad structure of the step-indexed fibre preform and the suspended core fibre design preform. The constructed fibre preform is drawn down to optical fibre using the fibre drawing tower. A wide range of fibre drawing parameters are used and tested out before we are able to successfully draw fibres from our preform.

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