Journal of Materials Processing Technology 210 (2010) 2016–2022

Contents lists available at ScienceDirect

Journal of Materials Processing Technology

journal homepage: www.elsevier.com/locate/jmatprotec

Extruded tellurite glass optical fiber preforms

Amit Belwalkar[a,b,*], Hongsheng Xiao[b,c], Wojciech Z. Misiolek[a,b,c], Jean Toulouse[d,e]

[a] *Department of Mechanical Engineering and Mechanics, Lehigh University, Bethlehem, PA, USA*
[b] *Institute for Metal Forming, Lehigh University, Bethlehem, PA, USA*
[c] *Material Science and Engineering, Lehigh University, Bethlehem, PA, USA*
[d] *Physics Department, Lehigh University, Bethlehem, PA, USA*
[e] *Center for Optical Technologies, Lehigh University, Bethlehem, PA, USA*

ARTICLE INFO

Article history:
Received 25 April 2010
Received in revised form 15 July 2010
Accepted 17 July 2010

Keywords:
Glass extrusion
Tellurite glass
Flow lines
Shear bands
Viscous flow
Holey fiber (HF)

ABSTRACT

The extrusion behavior of tellurite glass in the supercooled liquid region was investigated. Good extrusion formability was observed under low strain rates at various temperatures in the glass transformation region investigated. Tube and holey fiber (HF) preforms were fabricated from tellurite glass billets using a laboratory press. In particular, the results for three-spoke HF design and round tube preforms with composition $75TeO_2{\cdot}20ZnO{\cdot}5Na_2O$ (TZN) are presented. The extruded preforms with precise geometrical features, an excellent surface quality and no crystallization were achieved in the temperatures range from 344 to 360 °C and at ram speeds ranging from 0.002 to 0.01 mm/s. Discrete shear bands were observed in the preforms, increasing in number and/or becoming better defined with increasing load and ram speed. Fewer shear bands were present when increasing the extrusion temperature from 344 to 360 °C. Thus, subsequent extrudates were successfully fabricated free of shear bands, providing good optical homogeneity that yielded solid and holey fibers that could provide much improved optical performance.

1. Introduction

Tellurite glass exhibits unique optical properties, first and foremost good optical transmission in the mid-infrared, but also higher linear refractive and much higher nonlinear index and other nonlinear coefficients (Lin et al., 2009) and stronger and broader rare-earth emission (Marjanovic et al., 2003) than silica glasses. Most importantly, tellurite glasses possess better thermal stability and chemical durability than the chalcogenide glasses that have been extensively investigated. Finally, because they are soft, tellurite glasses are easier to form than silica. These properties make tellurite glass particularly attractive for mid-IR fibers with a variety of geometries, solid core/clad and microstructured, which can be used in fiber lasers and amplifiers for optical communications or fiber sensors for environmental and bio-medical applications.

Microstructured fibers containing air holes (holey fibers, HF) have attracted considerable attention recently because their geometry can be designed for desired dispersion and polarization properties, small confinement and enhanced nonlinearities, thus providing new functionalities compared to conventional core/cladding fibers according to Ebendorff-Heidepriem and Monro (2006). Thus, HFs appear promising for a number of applications in areas such as all-optical applications (broad band sources, amplification, switching, etc.) and power delivery. HFs are a single-material-based novel type of fibers with air channels arranged in specific patterns surrounding a solid or air core and running through the entire length of the fiber. HF preforms can be made using several techniques such as capillary stacking, drilling and casting, but all of these have certain limitations. Suction or rotational casting techniques are limited to simple geometries. Capillary stacking is laborious and time consuming and presents reproducibility problems, while drilling is limited to relatively short preform lengths. By contrast, extrusion offers great flexibility in preform and, ultimately, fiber geometry. It is a one-step, reproducible and versatile process that can conveniently produce complex cross-sections of different sizes, shapes and cross-section reductions. It is also favored over other bulk forming processes due to the advantageous state of stress when forming brittle materials like ceramics and glasses.

Silica HFs have now been produced with a variety of geometries. However, silica does not transmit light past 2 μm wavelength and new glasses and fibers are needed for mid-IR applications. Little work has been done so far on soft glass HFs in general and even less on soft glass HFs produced by extrusion and they are now attracting growing interest according to Monro and Ebendorff-Heidepriem (2006). Recently, a number of researchers successfully produced HF preforms of several glasses such as bismuth (Ebendorff-Heidepriem

---

[*] Corresponding author at: Department of Mechanical Engineering and Mechanics, Lehigh University, Bethlehem, PA, USA. Tel.: +1 4844325740.
E-mail address: aab306@lehigh.edu (A. Belwalkar).

0924-0136/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jmatprotec.2010.07.018

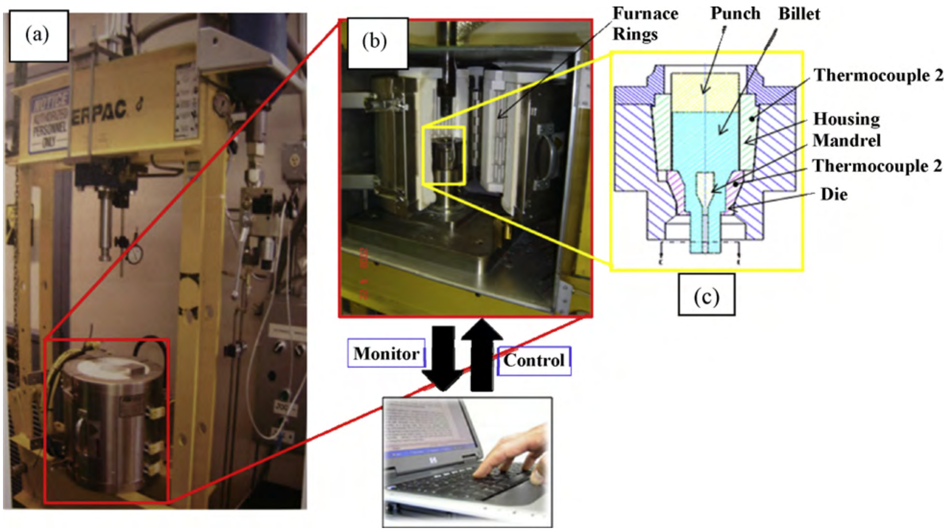

Fig. 1. (a) The laboratory vertical glass extrusion press and (b) extrusion press furnace and the tooling assembly (c) schematic of the die assembly.

and Monro, 2007), SF6 (Ravi Kanth Kumar et al., 2002), SF57 (Kiang et al., 2002; Petropoulos et al., 2003) as well as tellurite-based glasses (Ravi Kanth Kumar et al., 2003).

In the present paper, we report on the fabrication and testing of round tubes and three-spoke HF preforms from bulk tellurite glass by extrusion. The basic composition of the glasses considered was $75TeO_2{\cdot}20ZnO{\cdot}5Na_2O$ (TZN), because it possesses good formability and offers good optical properties. Other glasses of varying compositions were also extruded.

2. Experimental method

Tellurite glass was prepared by mixing 75 mol% TeO_2, 20 mol% ZnO and 5 mol% Na_2O powders that were carefully ground and well mixed into a fine powder. The powder mixture was dried at 200 °C for 1.5 h and the temperature was then raised to the melting temperature of 800 °C over 1.5 h and the melt held at that temperature for another 3 h. The billet was prepared by pouring the melt into a 32.5 mm diameter brass mold, which was kept on a brass plate preheated to 250 °C. The billet was then annealed at 300 °C for 7 h to remove residual stresses and then slow cooled to room temperature in the furnace.

Differential scanning calorimetry (DSC) was performed to determine the extrusion temperature range between the glass transition temperature, $T_g = 573$ K, and the onset of crystallization, $T_x = 680$ K, as reported by the authors (Belwalkar et al., 2010). Here we use degrees Kelvin for comparison with our previous work. The viscosity of the glass was also measured, using an advanced rheometric expansion system (ARES) in a parallel plate configuration between 609 and 663 K as a function of shear rate from 0.01 to 10 s^{-1}. As the glass sample was sheared, two different flow modes were observed, newtonian at lower shear rates and non-newtonian or shear thinning at higher shear rates. The extrusion could then be performed in the appropriate range where the glass can be viscously deformed without crystallization. Temperatures from 617 to 633 K and shear rates from 0.01 to 1 s^{-1} were determined as appropriate for extrusion of our glasses. Ram speeds were accordingly selected to vary from 0.002 to 0.01 mm/s.

Besides temperature and ram speed, the two other critical parameters in the glass extrusion process are the degree of friction and die geometry. Friction is mainly determined by the billet geometry, its surface quality and by the die design and die material. Friction between the supercooled glass and the die was minimized by cleaning and polishing the die thoroughly using selected polishing papers each time before extrusion to ensure a smooth finish of the tooling walls. Since the glass extrusion was carried out at high temperatures, a high temperature-resistant, high toughness Ni-based superalloy (Inconel) was selected as the tooling material. A die design concept known in literature as a porthole die (or bridge or spider die) was employed to minimize preform eccentricity in the round tube performs (DePari and Misiolek, 2010). The HF preform design is inspired by an earlier one proposed by Kiang et al. (2002) and Feng et al. (2005). This design is based on the index guiding principle and a simple microstructure having high index glass core and large air-filled holes surrounding it. The core guides the light and large air-filled fraction efficiently confines the light in the center core. The center core is held by three spokes having thickness 0.2 mm that also support the outside wall that confines the air holes. Due to such thin features as the spokes, the losses from the solid core could be negligible compared with the inherent losses from the material itself (Feng et al., 2005). For the design of HF perform and the die, refer to Fig. 3.

A vertical laboratory glass extrusion press was designed, fabricated and set up for this research as shown in Fig. 1a. It is equipped with a furnace for heating the glass billet (see Fig. 1b) and a motion controller connected to a computer for controlling the ram travel. The press maximum load is 300 kN and the ram can travel from 0 to 150 mm with a speed as low as 0.001 mm/s. The billet along with the entire die assembly, shown schematically in Fig. 1c, is placed inside the press furnace.

The temperature inside the furnace is controlled to the desired extrusion temperature by adjusting the temperature setting of three furnace rings. Two thermocouples are employed to measure the temperature in the extrusion die assembly, as shown in Fig. 1c, and to make sure that the extrusion temperature is maintained within the expected range. Once a stabilized extrusion temperature is achieved, the press begins to extrude the glass billet at the elevated temperature. The punch speed is accurately controlled during the extrusion process by BODAC software as per the experimental requirements. Typically, three sets of ram speeds are used. Depending on the height of the billet, the ram is allowed to accelerate until the punch gets very close to the top of the billet. The speed is then reduced to an intermediate level as soon as the punch touches the billet. Thereafter, the ram speed is reduced to the experimental speeds listed in Table 1 so as to maintain the requirement of a low strain rate in glass extrusion. The supercooled glass is