AFRL-AFOSR-JP-TR-2018-0073

High power durability of soft glasses for laser applications

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10/10/2018
Final Report

DISTRIBUTION A: Distribution approved for public release.

Air Force Research Laboratory
Air Force Office of Scientific Research
Asian Office of Aerospace Research and Development
Unit 45002, APO AP 96338-5002

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*
10-10-2018

2. REPORT TYPE
Final

3. DATES COVERED *(From - To)*
24 Jun 2016 to 23 Jun 2018

4. TITLE AND SUBTITLE
High power durability of soft glasses for laser applications

5a. CONTRACT NUMBER

5b. GRANT NUMBER
FA2386-16-1-4068

5c. PROGRAM ELEMENT NUMBER
61102F

6. AUTHOR(S)
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5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
THE UNIVERSITY OF ADELAIDE
115 GRENFELL ST
ADELAIDE, 5000 AU

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
AOARD
UNIT 45002
APO AP 96338-5002

10. SPONSOR/MONITOR'S ACRONYM(S)
AFRL/AFOSR IOA

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
AFRL-AFOSR-JP-TR-2018-0073

12. DISTRIBUTION/AVAILABILITY STATEMENT
A DISTRIBUTION UNLIMITED: PB Public Release

13. SUPPLEMENTARY NOTES

14. ABSTRACT
The overall aim of this project is the fabrication of high-quality mid-infrared glasses and fibers for systematic investigation of the high power laser durability of mid-infrared glass types at AFRL. The findings advance the knowledge of the chemical processes during raw material thermal treatment and glass melting, which provides guidance for future experimental and theoretical investigations of high-quality and reproducible glass fabrication. The project focused on heavy metal oxide glasses such as zinc-tellurite and lead-germanate as they exhibit a combination of attractive properties such as high rare earth solubility, high refractive index and high crystallization stability, and are therefore promising candidates for mid-infrared laser and nonlinear optical processing applications. The challenge for the fabrication of these glasses is to achieve low hydroxyl group content while preventing the formation of lower valency heavy metals such as metallic tellurium and lead as both hydroxyl groups and metallic species cause detrimental absorption in the glasses, diminishing performance in particular under high power laser conditions. Another challenge is the fabrication of core/clad fibers with superior interface quality in a process that is suitable for high degree of automation for future manufacture.
A new die design for dual-glass extrusion was investigated using zin-tellurite glasses. Using undoped zinc-tellurite core and cladding glass pair, the new dual-glass extrusion technique resulted in a unique combination of preform features such as complete fusion of the two glasses at the core/clad interface within the preform, small core/clad diameter ratio and consistent core diameter along the preform length. This combination of preform features is a significant advancement compared to the conventional rod-in-tube preform technique or co-extrusion of core and cladding

15. SUBJECT TERMS
Laser, High power, Durability, Soft glass, Mid-Infrared, Mid-IR, Flouride, Germanite, Tellurite, Scalability

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
Unclassified	Unclassified	Unclassified	SAR		SINGLETON, BRIANA

19b. TELEPHONE NUMBER *(Include area code)*
315-227-7007

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

Final Report for AOARD Grant FA2386-16-1-4068

"**High Power Durability of Soft Glasses for Laser Applications**"

23 September, 2018

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Period of Performance: 06/24/2016 – 06/23/2018

Abstract:

The overall aim of this project is the fabrication of high-quality mid-infrared glasses and fibers for systematic investigation of the high power laser durability of mid-infrared glass types at AFRL. The project focused on heavy metal oxide glasses such as zinc-tellurite and lead-germanate as they exhibit a combination of attractive properties such as high rare earth solubility, high refractive index and high crystallization stability, which makes them promising candidates for mid-infrared laser and nonlinear optical processing applications. The challenge for the fabrication of these glasses is to achieve low hydroxyl group content while preventing the formation of lower valency heavy metals such as metallic tellurium and lead as both hydroxyl groups and metallic species cause detrimental absorption in the glasses, which diminishes performance in particular under high power laser conditions. Another challenge is the fabrication of core/clad fibers with superior interface quality in a process that is suitable for high degree of automation for future manufacture.

The low processing temperatures of zinc-tellurite glasses made them an ideal model glass system to investigate the feasibility of a new die design for dual-glass extrusion and the diverse parameters affecting glass melt dehydration of a heavy metal oxide glass in a cost-efficient way. The second year of the project applied the knowledge gained in the first year of the project to the dehydration of lead-germanate glass, which promises higher laser durability due to higher glass transition temperature.

Using undoped zinc-tellurite core and cladding glass pair, the new dual-glass extrusion technique resulted in a unique combination of preform features such as complete fusion of the two glasses at the core/clad interface within the preform, small core/clad diameter ratio and consistent core diameter along the preform length. This combination of preform features is a significant advance compared to the conventional rod-in-tube preform technique or co-extrusion of core and cladding glass stacks, in particular for core and cladding glasses with similar viscosity.

The glass fabrication research comprised investigation of a large range of factors (~20 parameters) including process parameters, raw material purity, dehydration agents, melting atmosphere type and flow rate and melting facility parameters. This integrative approach to study glass melting behavior enabled understanding of the complex chemistry of the transformation of the solid raw materials into a glass melt and the complex interactions between the liquid glass melt, dissolved gas in the melt and surrounding gas atmosphere. This investigation also revealed the complex dependence of the melting atmosphere composition and moisture content on various sources of gas flow into and out of the furnace liner. For lead-germanate glass, the impact of all of these factors on the hydroxyl group content and presence of lower valence lead species was quantified for small melts of 15g. Application of the most suitable melting conditions to larger melt of 100g led to the fabrication of a low-loss glass billet of suitable size for fiber fabrication. The findings advance our understanding of the chemical processes during raw material thermal treatment and glass melting, which provides guidance for future experimental and theoretical investigations of high-quality and reproducible glass fabrication.

Direct laser writing experiments were undertaken in collaboration with Prof David Lancaster at the University of South Australia. Use of different repetition rate and pulse energy indicated the feasibility of writing channel waveguides into rare earth doped germanate glass, which paves the way towards chip laser development in infrared-transmitting heavy metal oxide glass.

Introduction:

The overall aim of this project is the fabrication of high-quality mid-infrared glasses and fibers for systematic investigation of the high power laser durability of mid-infrared glass types at AFRL. The project focused on heavy metal oxide glasses such as zinc-tellurite and lead-germanate as they exhibit a combination of attractive properties such as high rare earth solubility, high refractive index and high crystallization stability, which makes them promising candidates for mid-infrared laser and nonlinear optical processing applications.

Optical fiber fabrication comprises 3 steps: First a batch of crystalline raw materials is transformed to a glass via melting, casting and annealing. Secondly, one or more glasses are processed into a preform, which is in a third step drawn down to the final fiber. An alternative waveguiding structure fabrication technique is the direct femtosecond laser writing of channel waveguides into a glass slab. This project investigated the following aspects of glass melting, preform fabrication and waveguide writing.

Glass samples for AFRL

The project started with training of AFRL scientist Leanne Henry in the melting of tellurite and germanate glasses. This provided the opportunity to provide glass samples for collaborative research with Leanne early on in the project and allowed her to build her own glass melting laboratory at AFRL to expand future research. Details of the training are described below.

Glass dehydration

This task was motivated by the fact that reproducible fabrication of high-quality glasses with low content of detrimental species causing absorption and scattering is critical for the evaluation of the intrinsic durability of glass types under high power laser conditions. Hydroxyl (OH) groups in the glass show a strong absorption at 3-4 µm, which seriously diminishes infrared transmission. When melted in ambient atmosphere, heavy metal oxide glasses exhibit such high content of OH groups that they do not show infrared transmission over 0.01-1 m length scale. Therefore, minimizing the content of OH groups is critical for these glasses. Another challenge is the formation of lower valency states of polyvalent heavy metals such as metallic tellurium and lead. These species cause high absorption and scattering over the visible to infrared spectral region. Thus, the aim of this project was to understand the impact of a large range of factors on both OH group and metallic species content in zinc-tellurite and lead-germanate glasses.

We commenced with investigating reducing OH content in a glass melt, so called glass melt dehydration, using zinc-tellurite glasses. The low processing temperatures of zinc-tellurite glasses make them an ideal glass system to investigate diverse parameters affecting glass melt dehydration of a heavy metal oxide glass in a cost-efficient way. In addition, this allowed the project to build on previous experience in tellurite glass dehydration. The second year of the project applied the knowledge gained in the first year of the project to the dehydration of lead-germanate glass as this glass promises higher laser durability due to higher glass transition temperature. The comprehensive investigations of a large range of parameters for both zinc-tellurite and lead-germanate glass unraveled previously undescribed effects of interrelated parameters and their effects on glass melting behavior.

Core/clad preform and fiber fabrication

This task aimed to develop a reproducible and effective method for step-index core/clad preform fabrication with high-quality interface between core and cladding glass to overcome limitations of current preform fabrication techniques. To date, the majority of step-index soft glass preforms (including tellurite and germanate glasses) are made using rotational casting or rod-in-tube technique.

For rotational casting, the core and cladding interface is formed when the glass has low viscosity, i.e. when it is liquid, enabling good wetting between core and cladding glasses and hence high-quality interface between the two glasses without bubbles or other defects. However, rotational casting is a manual process requiring highly skilled operator and is challenging to scale up.

For the rod-in-tube technique, the small diameter core glass rod and the corresponding cladding glass tube are made separately using different techniques. The assembly of the core rod inside the cladding tube is used as the preform for fiber drawing. The core/clad interface is formed during fiber drawing when the glass is soft. Note that during fiber drawing, the glass is only within the hot zone of the furnace for a short time of 10-20 min, as the glass is continuously moved into and out of the hot zone. In previous work, we found that for core and cladding glasses with similar glass transition temperature, the rod-in-tube technique leads to imperfect fusion of the core and cladding glass during the short time heating of fiber drawing.

Previous research at the University of Adelaide led to a novel die concept for direct dual-glass extrusion into a step-index preform, which allows the core/clad interface to be formed under pressure during extrusion. This concepts promises high-quality interface, even for core and cladding glass combination where both glasses have similar glass transition temperature. The novel die concept relies on the separate fabrication of core and cladding glass billet. In this project, zinc-tellurite core and cladding glass combination of similar glass transition temperature was selected to investigate various methods for core and cladding glass billet fabrication with regard to time and cost efficiency and to compare the core/clad interface quality and loss in the fibers made using the dual-extrusion or rod-in-tube technique.

<u>Waveguide writing and laser development</u>

One way to test high power laser durability of a mid-infrared transmitting glass is to investigate power scaling of the laser operation of the glass. We selected waveguide chip architecture and lead-germanate glass doped with Holmium to develop a laser operating at ~2 μm wavelength. To date Ho^{3+} 2 μm laser are pumped with Tm^{3+} fiber laser at 1.9 μm. The use of a fiber laser for pumping is an uneconomical method. To overcome this issue, we selected to co-dope the lead-germanate sample with Ytterbium to allow pumping by readily available and inexpensive 980 nm diode lasers. After being pumped at 980 nm, we anticipate that Yb^{3+} will efficiently transfer phonon assisted energy from the Yb^{3+} $^2F_{5/2}$ level to the upper laser level Ho^{3+} $^5I_6$, thus enabling the use of laser diode for pumping. The advantage of the waveguide chip architecture is that direct femtosecond laser writing into the bulk glass can be used without the need for preform and fiber fabrication. This requires a gain medium with high rare earth concentration to compensate for shorter length. Lead-germanate glass is an ideally suited gain medium as it can be doped with high rare earth concentration and its higher thermomechanical stability relative to tellurite promises higher durability during laser writing and laser generation.

Experiment:

The experimental work was undertaken by two research assistants at the Institute for Photonics and Advanced Sensing (IPAS) at the University of Adelaide (UoA) and a PhD student at the Laser Physics and Photonics Devices Laboratory (LPPDL) at the University of South Australia (UniSA). The two research assistants were supervised by Prof Heike Ebendorff-Heidepriem at UoA and the PhD student was primarily supervised by Prof David Lancaster at UniSA.

<u>Glass fabrication:</u>

The glasses in this project were made using the state-of-the-art open air and specialized controlled atmosphere glass melting capability at IPAS. The glass fabrication comprised:

1. glass batch preparation inside a glovebox,
2. heating the glass batch inside a furnace to elevated temperature, resulting in complete melting and dissolution of all raw material components,
3. casting the glass melt into a pre-heated brass mold,
4. annealing of the glass in the mold at the glass transition temperature and slow cooling down to room temperature.

The following fabrication parameters were employed:

Glass composition: The two tellurite glass compositions and the germanate glass composition allowed the impact of glass viscosity and melting temperature to be investigated.

Raw material batch: For the major tellurite glass component TeO_2, we investigated the glass melting behavior of raw materials from different suppliers. The two raw material batches investigated differed in weight loss, particle size and volume.

Melting temperature and time: The melting procedure of the glass batches is specified according to the temperature of the furnace at the crucible location and the total time from placing the crucible into the furnace to taking the crucible out of the furnace for casting. When placing the crucible into the furnace at a lower temperature than the final melting temperature, the heat up rate was also considered. The final melting temperature for tellurite and germanate glasses was ~800 ºC and 1250 ºC, respectively. Melting times ranged from 60 min to several hours.

Melting atmosphere: The glasses were either melted in open air furnace under ambient atmosphere, i.e. in air with >1000 ppm moisture, or in a furnace attached to a glovebox box and purged with dry nitrogen or oxygen or mixture of the two gases containing ~1 ppm moisture or less. For germanate glass, different gas flow rates into the furnace liner as well as different glovebox pressurization was used to understand impact of gas flow dynamics inside the furnace liner where the crucible was located.

Glass batch weight and crucible volume: The weight of the glass batches was in the range of 15-100 g. The tellurite glass batches were melted in gold crucibles of 100 mL or 300 mL volume. The germanate glass batches were melted in platinum crucibles of 100 mL or 200 mL volume. Different combination of raw material batch, glass batch weight and crucible volume allowed investigation of the impact of the surface-to-volume ratio of glass batch and glass melt.

Glass batch dehydration step: For a selection of glass batches, we investigated the impact of a dehydration step for the batch before melting the batch in order to explore dehydration efficacy for batch and melt.

Dehydration agent: For the germanate glass, we investigated use of chloride salts in the glass batch on the efficiency to remove hydroxyl groups from the glass batch and glass melt.

Casting and annealing parameters: For the dehydration investigations, a rectangular brass mold and fixed pre-heating temperature and annealing procedure was used. For the core and cladding glass billet fabrication, different mold designs were investigated. Preheating temperature and annealing procedure was adjusted to the mold design used.

For investigation of dehydration and for waveguide chip development, small glass blocks of $30 \times 15 \times (5\text{-}15)\ mm^3$ were prepared. For dual-glass extrusion, glass billets of 30 mm diameter were prepared.

Glass characterization:

The glass samples made for glass dehydration study were characterized using commercial transmission spectroscopy instrument at IPAS at UoA. The absorption intensity of the fundamental vibration of the OH groups at ~3 µm served as measure for the OH content in the glass. The position of the UV edge served as a measure for the presence of metallic lead species in the lead-germanate glass made. In addition, coloration of batch and glasses indicated the presence of metallic tellurium and lead species in batch and glass.

To assess laser performance potential of the fabricated lead-germanate glass, the absorption and emission cross-sections were measured using commercial transmission and fluorescence spectroscopy instruments at IPAS and LPPDL.

Glass extrusion, fibre drawing and fibre characterisation:

All the experimental work was done at IPAS. The dual-glass extruded preform was made by joint extrusion of the core and cladding glass billets through the die with novel design into a preform where core and cladding glass were fused together. The rod-in-tube preform was made by first extruding a 10 mm diameter rod from the core glass billet and a 10 mm outer diameter tube from the cladding glass billet. The 10 mm core glass rod was scaled down to a thin, ~2 mm diameter rod using the soft glass draw tower. Finally, the thin core rod was inserted into the cladding tube, forming the assembled rod-in-tube preform. Both the dual-glass extruded preform and the rod-in-tube preform were drawn down to fibers of 160 µm outer diameter.

The core diameter along the length of the dual-glass extruded preform was determined using a coordinate-measuring machine, which allowed non-destructive measurement. The cross-sectional geometry and the core/clad interface of the fibers were measured using images taken with scanning electron microscope (SEM). The propagation loss of both fibers was measured using broadband white light source and cutback method.

Laser writing of channel waveguides:

Single line bidirectional waveguides were written into the germanate glass at the LPPDL femtosecond laser machining facility using both 5 MHz and 1MHz repetition rate pulse trains over a range of pulse energies.

Results, Discussion, Future Plans:

Glass samples and training for AFRL; future collaboration

Two months of the first year were dedicated to train Leanne Henry from Kirtland AFRL in the fabrication of oxide glasses using the melt-quench technique. Assisted by experienced staff, she operated both the open air glass melting facility and the specialized controlled atmosphere glass melting facility at IPAS. Leanne prepared Bi-, Er- and Er:Yb-codoped tellurite and germanate glasses for future investigation of the laser properties of these samples at the laser labs at AFRL. Leanne was also shown the principle of billet extrusion and fiber drawing for tellurite glasses of relevance to this project. A Material Transfer Agreement for sending the glass samples to AFRL has been signed and the samples arrived at AFRL in November 2017. The samples were used as references to evaluate the quality of the samples made by Leanne at AFRL after she has set up an open air glass melting facility at AFRL in 2017 based on the knowledge in glass melting technology she gained at IPAS. Leanne Henry (AFRL), Heike Ebendorff-Heidepriem (IPAS) and Ravi Jain (University of New Mexico) have had discussions on future collaborations, which led to Ravi Jain applying for Fulbright Scholarship to visit IPAS for a few months in 2019/2020. Currently, we are discussing concrete plans for collaborative research on novel tellurite and germanate glass lasers.

Glass dehydration

First analysis of the various experimental results indicates that the OH content in tellurite and germanate glasses is not only sensitively affected by the fabrication conditions used for the liquid glass melt, but also by the raw material powder properties (e.g. particle size) and the specific conditions of the thermal processes to completely transform the solid raw materials into a homogeneous glass melt.

Systematic and in-depth investigations of lead-germanate glasses revealed the sensitivity of OH content on the moisture content of the gas in the furnace liner, i.e. around the crucible containing the glass melt. The moisture content in turn was strongly dependent on different gas sources entering the furnace liner as the furnace liner is not hermetically sealed from the environment and thus not only the controlled dry gas purge into the liner but also ultra-dry nitrogen from the glovebox or backflow from ambient wet atmosphere can take place. Furthermore, the OH content depends on the gas type. Nitrogen is considerably more effective in OH content reduction, however, it led to the formation of detrimental metallic lead species. Thus, we investigated the balancing of oxygen and nitrogen content in the furnace liner to achieve best compromise of low OH content and absence of metallic lead in the glass.

The use of various chloride salts for dehydration demonstrated the importance to take into account the decomposition temperature of the salt. We also found that the dehydration efficiency of a salt depends on at which time and temperature the chloride salt is added to the batch or melt.

Considering all knowledge gained, we successfully upscaled the glass melt volume from 15 g to 100 g, while maintaining low OH content and preventing formation of metallic lead. The billet cast from a 100 g melt is sufficiently large for extrusion and fiber drawing in the next few months.

The experimental results and observations indicated the complex nature of the chemistry of the interaction of the atmosphere and chloride salts with the melt or batch. Some of the results have challenged our current understanding of the process chemistry, leading to new theory about the effect of hitherto unexplored parameters, which provides guidance for future experimental and theoretical investigations of high-quality and reproducible glass fabrication. The findings will be described in detail in two papers.

In the near future, we will seek funding to apply the knowledge gained for tellurite and germanate glass dehydration to the fabrication of rare earth doped heavy metal glasses to be used for step-index and microstructured fiber fabrication for mid-infrared laser and supercontinuum generation. In addition, we plan to use our knowledge on the redox chemistry of heavy metals in glass to investigate fabrication of glasses with controlled redox state for bismuth, tellurium or lead for broadband emission in the infrared.

Core/clad preform and fiber fabrication

For the fabrication of the small diameter core glass tellurite billet (i.e. ~6 mm rod of ~50 mm length), we evaluated extrusion and direct casting. While previous research has demonstrated the reproducible extrusion of 10 mm diameter tellurite rods in high optical quality, a first extrusion trial of 6 mm diameter tellurite rod indicated the need for identification of suitable extrusion conditions for small diameter rod. As the direct casting of a small diameter rod is a more time and material effective method compared with extrusion, we explored direct casting in detail. Optimization of the casting mold design resulted in a small diameter rod, i.e. core glass billet, with high optical quality.

For the fabrication of the doughnut shaped cladding glass tellurite billet, we evaluated drilling and direct casting. Drilling of the hole into a solid billet was found to result in cracking of the glass. Investigation of different casting mold designs and annealing procedures eventually led to the fabrication of a high quality doughnut shape billet.

These results demonstrate that the time-effective method of casting is a promising method for the fabrication of both small diameter core glass billet and doughnut shaped cladding glass billet for direct extrusion of step-index preform.

The cast core glass billet and cladding glass doughnut shaped billet were used for dual-glass extrusion through a metal die. The preform demonstrated fusion between core and cladding glass and a core/clad diameter ratio of 0.2, which agrees with the die design. The preform was drawn down to fiber of 160 μm outer diameter, resulting in ~30 μm core diameter.

Using the same core and cladding glass composition, a rod-in-tube preform assembly was prepared using extrusion and drawing as described above. The preform was designed to have also a core/clad ratio of 0.2 as for the dual-glass extruded preform. However, the extruded tube had smaller hole size than expected, resulting in the final fibre to have core/clad ratio of 0.15.

SEM images of the both the dual-glass extruded fiber and the rod-in-tube fiber showed the presence of bubbles at the interface for the rod-in-tube fiber, but the absence of bubbles at the interface for the dual-glass extruded fiber, indicating complete fusion between core and cladding glass for the dual-glass extruded fiber. This fiber showed loss almost as low as a comparable unstructured fiber, while the rod-in-tube fiber exhibited ten times higher loss. These results demonstrate that dual-glass extrusion enables fabrication of step-index fibers with high quality of the core/clad interface, which is critical for achieving low loss.

In the near future, we will seek funding to apply the dual-glass extrusion technique to a core and cladding zinc-tellurite glass combination, where the core glass is doped with erbium to fabricate a doped tellurite fiber that allows investigation of laser operation and therefore laser power durability. Furthermore, we plan to use the dual-glass extrusion technique to fabricate step-index doped germanate glass fiber for mid-infrared laser operation.

Chip laser development

In this project, glass characterisation and waveguide writing experiment were conducted as first steps towards germanate chip laser development.

The emission spectrum of the Ho^{3+} doped lead-germanate glass sample showed three peaks. The strongest emission cross of ~ 6×10^{-21} cm^2 was found for the 2.04 μm transition, confirming its suitability for the development of a laser around this peak in the short-wave mid-infrared region. The lifetime of the upper laser level was measured to be 6.7±0.03 ms, reflecting its potential to be an efficient candidate as a laser.

Single line bidirectional waveguides were written into the germanate glass using both 5 MHz and 1MHz repetition rate pulse trains over a range of pulse energies. Both repetition rates produced waveguides which were formed due to accumulation of heat surrounding the focal volume. The modified region displayed a 'tear-drop' elliptical shape due to the focal spot suffering spherical aberration when focused into the high refractive germanate glass. Focusing the laser writing beam deeper into the glass resulted in more severe spherical aberrations, leading to further elongation of the waveguides. We also investigated a lower repetition rate of 100 kHz, however it resulted in more elliptical waveguides which were founded to have higher propagation loss. We concluded that the waveguides formed by the 5 MHz repetition rate are more suitable to realize low-loss waveguides at writing powers <400 mW. Increasing the femtosecond laser power above this value resulted in cracked waveguides.

The resulting waveguides displayed multimode characteristics with a numerical aperture of 0.07 ±0.02 and a positive refractive index contrast of 0.9×10^{-3}. The waveguide loss was determined to be ~1.5±0.6 dB at 633 nm. These results demonstrate the ability to write low-loss waveguides in lead-germanate glass.

In the future, we will investigate the Yb-Ho energy transfer and conduct laser experiment at ~2 μm for laser written channel waveguides.

Publication plan:

We plan to submit the following papers in the near future.

- Dec 2018: Development of low-loss lead germanate glass for mid-infrared fiber optics: Part I - Glass melting;
- Mar 2019: Development of low-loss lead germanate glass for mid-infrared fiber optics: Part II - Fiber fabrication and characterisation
- Mar 2019: New dual-glass extrusion technique for making step-index fibers
- Jun 2019: Direct laser written waveguides in Yb:Ho-doped lead-germanate glass

Significant Collaboration that resulted from your AOARD supported project:

This project significantly supported collaboration with laser scientist Leanne Henry from AFRL at Kirtland Air Force Base. Two months of the first year were dedicated to train Leanne in the fabrication of oxide glasses using the melt-quench technique. Assisted by experienced staff, she operated both the open air glass melting facility and the specialized controlled atmosphere glass melting facility at the Institute for Photonics and Advanced Sensing at the University of Adelaide. Leanne prepared Bi-, Er- and Er:Yb-codoped tellurite and germanate glasses for future investigation of the laser properties of these samples at the laser labs at AFRL. Leanne was also shown the principle of billet extrusion and fiber drawing for tellurite glasses of relevance to this project. A Material Transfer Agreement for sending the glass samples to AFRL has been signed and the samples arrived at AFRL in November 2017. Leanne Henry has used these samples as references to evaluate the quality of the samples made by her at AFRL after she has set up an open air glass melting facility at AFRL in 2017 based on the knowledge in glass melting technology she gained at IPAS.

Leanne Henry (AFRL), Heike Ebendorff-Heidepriem (IPAS) and Ravi Jain (University of New Mexico) have had discussions on future collaborations, which led to Ravi Jain applying for Fulbright Scholarship to visit IPAS for a few months in 2019/2020. This Fulbright project at IPAS will focus primarily on the development of advanced mid-IR glasses and the use of these glasses to make optical fibers and the testing of critical optical properties of these fibers at various external labs vis-a-vis their performance in fiber lasers and microlasers. This would further strengthen the collaborative research between AFRL, University of New Mexico and University of Adelaide. In addition, Heike Ebendorff-Heidepriem plans to visit Leanne Henry at AFRL in 2019 to foster transfer of knowledge in glass fabrication and determine the scope for future joint research program.

Attachments: none