Laser heated pedestal growth system commissioning and fiber processing

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ABSTRACT

A new Laser Heated Pedestal Growth system was designed and fabricated using various aspects of effective legacy designs for the growth of single-crystal high-temperature-compatible optical fibers. The system is heated by a 100-watt, DC driven, CO_2 laser with PID power control. Fiber diameter measurements are performed using a telecentric video system which identifies the molten zone and utilizes edge detection algorithms to report fiber-diameter. Beam shaping components include a beam telescope; along with gold-coated reflaxicon, turning, and parabolic focusing mirrors consistent with similar previous systems. The optical system permits melting of sapphire-feedstock up to 1.5mm in diameter for growth. Details regarding operational characteristics are reviewed and properties of single-crystal sapphire fibers produced by the system are evaluated. Aspects of the control algorithm efficacy will be discussed, along with relevant alternatives. Finally, some new techniques for in-situ processing making use of the laser-heating system are discussed. Ex-situ fiber modification and processing are also examined for improvements in fiber properties.

Keywords: Laser-heated pedestal growth, sapphire optical fiber, harsh-environment sensors, fossil energy

1. INTRODUCTION

In the past decade or more, the National Energy Technology Lab (NETL) has sponsored several research projects in the development of high-temperature optical sensors for power and energy applications. These projects have resulted in the realization of novel high-temperature fibers, as well as sensing elements necessary to perform in the high-temperature regime. This Crosscutting Research Program has aided in the discovery of micro-structured sapphire fibers [1], spinel claddings [2], interferometric sensors [3], and numerous other innovations. Generally speaking, operation above about 1000°C requires the manufacture and implementation of single-crystal optical fiber materials which have higher melting and softening points than standard silica fibers. Furthermore, some single-crystal materials have shown superior resistance to degradation pathways such as OH diffusion or infiltration by other species in the process environment. These durability features, in conjunction with the infrared (IR) transmission properties of select materials, have spurred considerable research in the harsh-environment fiber field.

Today, sapphire (single-crystal Al_2O_3) fibers have gained considerable traction as one of the most robust platforms for high-temperature optical sensors. The most common method for the production of sapphire optical fibers is the Laser Heated Pedestal Growth (LHPG) system pioneered originally by Burrus and Stone [4], Harrington et al at Rutgers [5], Fejer et al at Stanford [6], and others. Thorough reviews of the method have been published previously [7, 8]. The LHPG technique is in principle rather simple. A rod or pedestal of oxide feedstock is heated by an IR laser to produce a molten oxide pool on the vertical pedestal's tip, while a single-crystal seed of the desired orientation is lowered into the molten pool. As the seed crystal is withdrawn vertically, a single-crystal fiber is grown by pulling the seed upwards continuously. The growth rate tends to be rather slow - on the order of a few millimeters per minute for most fibers. CO_2 lasers have traditionally been used for the laser-heating source at 10.6 µm due to strong absorption of that wavelength by the sapphire or alumina pedestal material. As little as a few tens of watts of laser power is sufficient to melt pedestals with diameters ranging up to about 1 mm. Because the process occurs at the focus of the CO_2 laser beam, the process tends to be sensitive to power variations and beam shape. Therefore, in order to provide uniform circumferential heating of the molten zone, a Gaussian beam-shape is often transformed into a "doughnut" beam using a reflaxicon optic [9]. Various mechanical systems have been used to ensure the precise upward motion of the pedestal along with the quicker upward travel of the seed and grown fiber. To date, high-quality unclad sapphire optical fibers can be purchased commercially in small quantities, but at costs more than several thousand times the price of standard silica fibers.

Although bare sapphire fibers are commercially available, there is still a significant need for further laboratory development of other single-crystal fiber platforms, processes for lowering the cost of growing fibers, techniques for sensor production in-process, and for methods of cladding application during or after fiber production. These developments will further aid in the commercialization of single-crystal fibers, fiber-sensors, and other devices. Given these technological needs, we designed and constructed a new LHPG system to investigate single-crystal fiber development in conjunction with our harsh-environment sensor program. The system has been constructed using design features purported to be the most successful in terms of operational characteristics. Consideration was given to future modification of the system in light of process improvements and the insertion of new techniques and methods as they may arise.

2. LHPG SYSTEM DESCRIPTION

The NETL LHPG system is diagrammed in **Figure 1**. A Parallax Technologies PLX-100s, 100 watt CO_2 laser outputting 10.6µm wavelength with an m^2 beam quality of 1.1 is used as the laser source. This laser was chosen because it exhibits extremely high power stability (in the 1% regime after 10 minute warmup). The high power stability is a result of the unique high-voltage DC tube operation, which differs from the standard industrial laser method of RF plasma excitation. The CO_2 beam is routed through a set of beam tubes with gold mirrors exiting in a ZnSe beam expander which provides expansion of the IR beam from about 7mm ($1/e^2$ beam diameter) to about 28mm, for 4X expansion. The second lens in the 2-lens beam expander is translatable via a sliding mount to provide for perfect collimation or slightly divergent output beams. The expanded beam is then directed through a gold-coated diamond turned reflaxicon (Nipro Optics). The beam first encounters the reflaxicon's inner core mirror which forms a back-reflected expanding doughnut beam. The back reflected beam is then collimated by the second reflaxicon cone. The smaller conical mirror is affixed to a 4" (102mm) diameter, ¼" (6.4mm) thick, ZnSe flat window so that there is no mounting hardware to create a shadow in the doughnut beam output. A 45-degree gold-coated turning mirror directs the beam upward towards a 4" (102mm) diameter, 3" (76mm) focal length parabolic focusing mirror which focusses the beam downward onto the pedestal material. The optical train was devised as to produce a molten zone which is uniform over the entire 360-degree axial direction with no shadows or dark spots present in the laser-focus. This ensures uniform circumferential heating of the molten spot, which aids in the uniformity of the fibers produced [5].

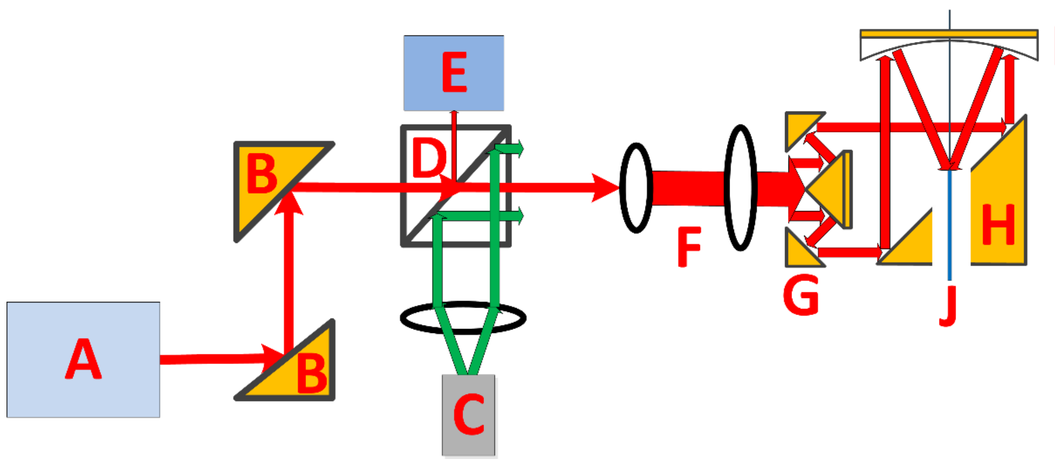

Figure 1. Optical system diagram. A: CO_2 laser, B: Gold flat mirrors, C: Alignment diode laser, D: Beam combiner, E: Power meter, F: Beam expander, G: Reflaxicon, H: 45-degree turning mirror with central hole, I: Parabolic focusing mirror with central hole, J: Pedestal rod

The mechanical system responsible for fiber pulling is shown in **Figure 2**. Therein, a set of precisely machined stainless steel v-groove blocks were fabricated to support and position fibers and pedestals with diameters ranging from 1mm down to 100µm. A set of urethane drive-belts were specified to pull the fiber or pedestal upward while in contact with the v-grooves. A three-pulley system was used to drive the belts with one motor pulley and 2 idler pulleys. The idler pulley directly below the v-groove is fixed along with the motor pulley, while the third pulley can be translated to tension the urethane drive belt. A belt-backing block which can also be translated is used to push the belt and fiber towards the v-groove during operation. The entire belt assembly can also be translated to provide controlled pressure between the belt and v-groove block. This system of belt-tensioning and variable fiber pressure was designed to accommodate a wide range of fiber materials and diameter ratios with similar performance over that entire range. This system also eliminates additional speed reduction components by using a highly accurate set of brushless, slotless, DC servo motors with 1000 line incremental amplified sine wave encoders (Aerotech BSM100). The motor system drives the belt directly with a 0.625" (16mm) diameter drive pulley to eliminate irregularities generally encountered with gearboxes or other mechanical speed reduction mechanisms. Together, the belt and motor drive system permit accurate control of fiber and pedestal motion.

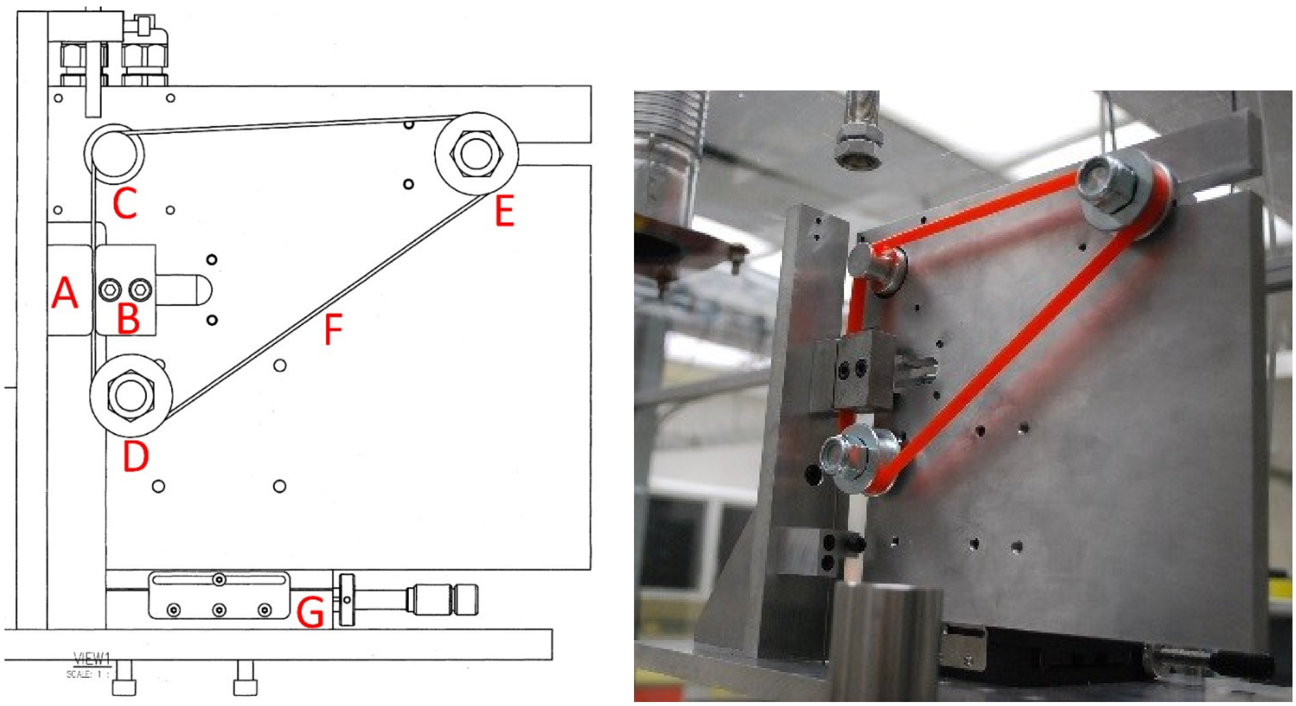

Figure 2. Schematic (left) and Photo (right) of the fiber pulling mechanism. A: V-groove block, B: Belt-backing block, C: Motor drive pulley, D: Fixed free-wheeling pulley, E: Tensioning pulley, F: Urethane belt, G: Belt system translator stage

Many publications have addressed controlling the diameter of fiber grown with a LHPG system. Previous researchers have used laser measurement systems [5] and visible or near-IR camera-based measurements [10] to carefully monitor the fiber's diameter and provide feedback to control LHPG operation. The NETL LHPG system employs two different diameter measurement techniques to further studies in diameter control and uniformity. The first system employed is a visible light camera system which observes the incandescence of the molten material. An Edmund Optics (EO-1918C) 1/1.8" CCD camera is used for imaging the molten zone. The camera is based on a 1600 X 1200 pixel array with sensor dimensions of 7.04mm X 5.28mm which is capable of producing between 2 and 8 frames per second in the current configuration. High quality measurement images are produced with a set of three compact in-line telecentric lenses. All three lenses have focal lengths of 65mm but possess magnifications of 1X, 4X, and 8X respectively. The different magnification lenses are used to appropriately fill the video frame with varying pedestal sizes and diameter ratios. The second measurement device is a Mitutoyo Laser micrometer (LSM-6200). It operates at 650nm to produce a shadow-graph type measurement of diameter with a better than 1μm accuracy. Similar accuracy is achievable using the camera-based system. The video system provides the additional benefit of monitoring the shape of the molten zone to correct for any misalignments during operation.

Presently, both the camera-based and laser scanning micrometer-based fiber diameter measurements are collected and processed using National Instruments (NI) Labview software, with additional signals and information being collected using an NI USB data acquisition system (NI-6211). To facilitate automation of most functions, the data acquisition system collects information on the cooling water temperature and laser power, while also producing a power-control voltage signal for the laser. Measurement of the laser power is accomplished by splitting off a portion of the CO_2 laser beam and directing it towards a power meter (Thorlabs PM100D, S310C sensor). Both the splitting of laser power for measurement and the co-linear introduction of a guiding red alignment laser is accomplished using a CO_2 laser beam combiner (II-VI 394265) which is highly reflective for the 635nm alignment laser diode while being highly transmissive for the 10.6μm beam (see Figure 1). The beam combiner reflects about 1% of the total laser power towards the power meter, so no additional beam splitting is required. A simple PID control loop is implemented in Labview which samples the laser power every 100ms and updates the power-control voltage signal. It is possible to achieve better than 0.5% power stability using this system.

In order to evaluate different methods of fiber-diameter control, a set of three primary algorithms were constructed to interface between incoming diameter measurements and system operations using several known diameter adjustment methods. A screen-shot of the operating software is shown in **Figure 3**. Therein, video output of the raw image, processed intensity image being measured, and plots of grown diameter and laser power are shown. The program has controls for motor operation functions, image processing parameters, and laser power. All three diameter-control algorithms are based upon simple proportional/integral/derivative (PID) calculations, but act upon different output parameters. The first algorithm utilizes fixed drawn-fiber and pedestal rates of movement, while varying laser power to adjust the diameter. When laser power is increased, the temperature of the molten pool increases, and the viscosity of the molten pool decreases, which results in smaller diameters. This method works relatively well, although it is necessary to carefully determine the appropriate ratio of pedestal movement rate and fiber pulling rate to achieve a particular diameter reduction ratio which is then fine-tuned by the applied power. The method is inherently hampered by requiring frequent changes in laser power. Rapid changes in laser power generally result in poor power stability at any level which make this method less desirable for high-reproducibility. The second and third algorithm both utilize constant controlled laser power along with one constant movement rate for either the pedestal or grown-fiber, along with PID control of the other movement rate. We note that the second and third algorithm are basically identical, although we find it easier to draw fiber with a constant rate and use PID control on the pedestal motion. In either case, it is necessary to adjust the PID constants for every combination of feedstock and drawn-diameter ratio leading to a plethora of "growth recipes" for different fibers. Furthermore, the quality of a given fiber will be a product of the algorithm applied, the PID constants employed, and the intrinsic stability of the system.

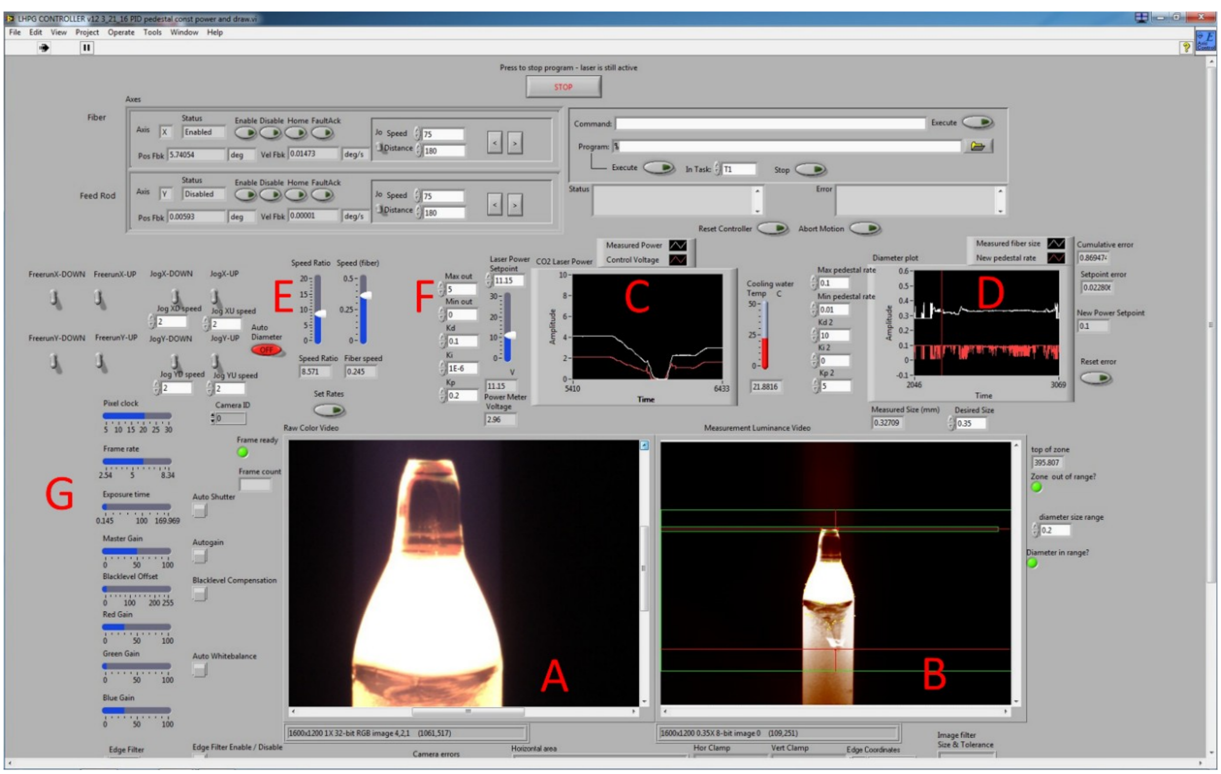

Figure 3. Operating software screen-shot. A: Original molten-zone image, B: Processed measurement image, C: Laser power and control signal plot, D: Diameter and pedestal control plot, E: Fiber speed and motor controls, F: Laser power controls and PID constants, G: Camera acquisition controls

3. GROWTH AND EVALUATION OF INITIAL SAPPHIRE FIBER SAMPLES

In order to evaluate the performance of the new NETL LHPG system, initial reductions of macro-sized sapphire feedstocks were performed. Sapphire rods were obtained from Insaco Inc. with diameters of 1 mm and 1.5 mm, all 100 mm in length. A 3:1 reduction ratio would then result in a ~900 mm long fiber. Diameter tolerance of 50 µm was specified for these pedestal feedstock rods. The feedstock and the fiber seed had their c-axis oriented vertically along the growth axis. Growth was performed in air using 25-50 watts of applied laser power for the ~3:1 reduction using the 1 mm rods. The limit for the maximum diameter of a pedestal which can be melted uniformly with full laser power was determined to be slightly less than 2 mm. Several pieces of fiber were produced in 10-30 cm lengths to evaluate the performance of the system. One such fiber is shown in **Figure 4**. The fiber grown was optically clear to the naked eye and under microscope examination. Good directional stability of the growth process resulted in the absence of bends or angles in the fiber. Some variation in diameter can be seen, which was later measured and reported here. Optical transmission measurements were not yet performed, although the loss in pure single-crystal sapphire fiber is expected to be between 2dB/m and 1dB/m at visible wavelengths, decreasing towards longer wavelengths. The production of these test sections permitted further refinement and improvement to the LHPG system which will be discussed here as well.

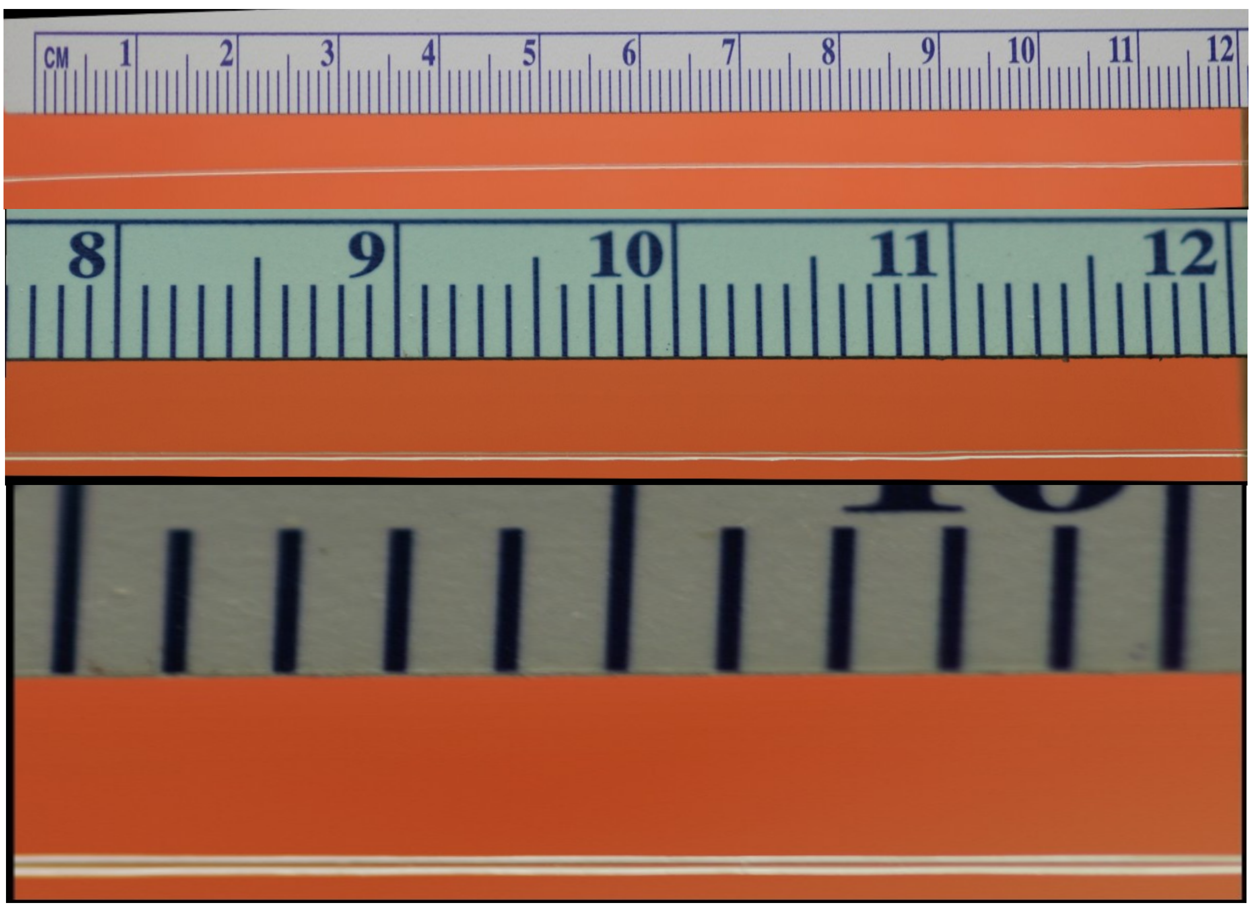

Figure 4. Picture of initially grown 350 µm diameter sapphire fiber at three magnification levels

To evaluate both the stability of the system in addition to the efficacy of the control algorithm, sections of initially grown fiber were re-measured using the video measurement system, but with no applied laser heating. A small incandescent backlight and a change in integration time permitted clear imaging of the grown fiber without the incandescence from the laser heating. The fiber was drawn upward at a rate of about 160 µm/s with about 2.5 frames-per-second (fps) camera capture yielding one diameter measurement every 64 µm along the length of the fiber. The overall measurement accuracy of the system is somewhat difficult to state since there can be small variations in measurements based on applied lighting conditions and any small vibrations of the fiber present during the measuring operation. When measuring small fixed unheated samples with 1X magnification, we generally find diameter measurements to be accurate within 2 µm, with better than 1 µm accuracy for the larger magnification lenses. **Figure 5** shows the measured diameter of the fiber pictured in Figure 4. It was noted that a few frames produced erroneous measurements which were discarded because too large a diameter change over a small length is physically impossible, and was not observed by the operator. This feature permits the software control algorithm to reasonably omit measurements that are more than 50 µm or so different from the previous measurement. Naturally, these fast variations will only occur during a high-angle taper or at the end of the fiber. It was determined that supporting the end of the grown fiber so that it does not swing due to air currents while being re-measured eliminates most erroneous measurements, although this generally requires fixing an additional section of fiber to the end of the grown section. Figure 5 shows the diameter measurements for a fiber grown with a diameter setpoint of 350 µm. The variation in diameter is about +/-10%. While this level of variation may be a bit higher than desired for the fiber, it is indicative of the initial capability of the LHPG system. It is also noted that during startup periods (not shown here), the diameter exhibits some long-period oscillation about the operating point, indicating the need for further tuning of the diameter-control PID constants. While this is a rather straight-forward proposition involving increasing the proportional constant to produce intentional oscillation and then calculating the appropriate integral and derivative terms, the process is rather time consuming given the need to establish steady state operation with each new set of constants being evaluated. We also note a small absolute deviation between the diameter set-point and the mean diameter produced. This is likely the result of camera detector saturation resulting in slightly larger measured diameters during the growth process. This effect can easily be offset in the software. Given the existing operational characteristics reported and the ability to further refine the operating algorithms, we feel that operation within 1% diameter variation for most fiber sizes will be possible in the present system. Further improvements in diameter variation will likely require mechanical modification of the system.

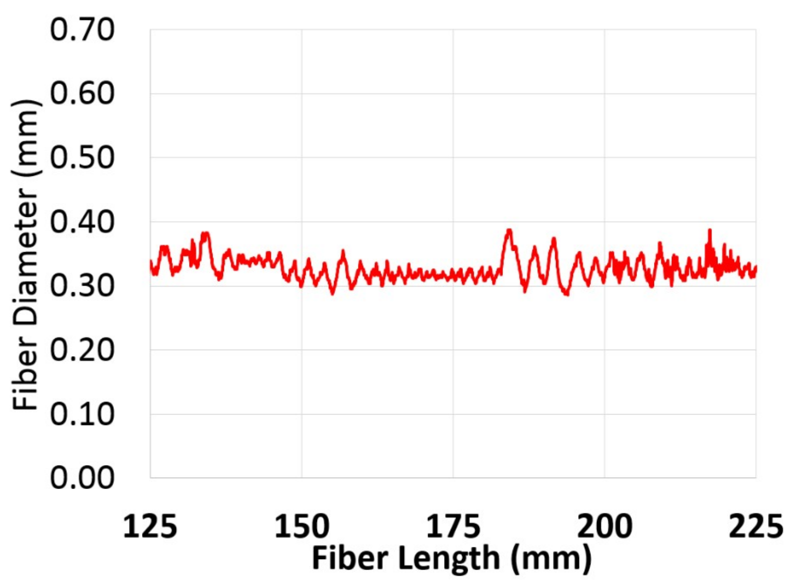

Figure 5. Example measured fiber diameter over 10cm with a 350µm fiber

4. OVERALL SYSTEM EVALUATION

While the LHPG process has been known for more than 40 years, the physical construction and implementation of a new system proved both technically challenging and complicated. The wealth of existing literature on the subject made available by several notable groups pioneering the technology made it possible to incorporate the most fruitful design concepts into the new system. Although some of the mechanical and optical components were custom fabricated, most of the system was constructed using off-the-shelf components and standard hardware. Although many operational details on existing systems were readily available, there were still numerous engineering decisions to be made in order to complete our design. We will briefly summarize some of the intended changes or modifications we believe will further improve the operation of our system to achieve superior operational characteristics.

The first item to address improving is the mechanical drive system constructed here. The system utilizes a urethane drive belt to move the fiber and pedestal, although the belt is of considerable length (about 19” (48.3cm) circumference). While drive motion of the belt systems is smooth and even, the belt itself will always exhibit some stretching. This causes a small delay between applied movement commands and actuation. Given the extremely slow rate of fiber growth, this delay is not a huge problem, but does affect the diameter uniformity somewhat. The problem is more pronounced when considering the pedestal motion via the same belt-drive system. Using the pedestal-PID algorithm, we find that the belt backlash when reversing direction is significant and can destabilize growth if the pedestal is permitted to travel in the downward direction to reduce the diameter. This intrinsic mechanical limit permits a range of pedestal motion from completely stopped (not moving upward) to some maximum upward velocity determined by the fiber-pulling rate. While stable growth can be achieved with this range of motion, the length over which the fiber diameter returns from an over-shoot is increased, and the overall diameter variation is therefore increased. We suggest a slight modification to permit increased control-stability. Removing the 3-pulley system and moving to a single urethane-coated pulley should remove all of the system backlash and permit faster actuation of fiber movements. While the contact patch would be reduced between the fiber and urethane, we do not expect significant slippage between such a pulley and the fiber or pedestal. Secondly, if redesigning the belt system, it would be beneficial to reduce the distance between the molten zone and v-groove to make alignment simpler.

The next element to be improved is the control of the laser-spot location. **Figure 6** shows the device used to hold the sapphire pedestal rod. A double-ended collet chuck grips the pedestal at one end, and a glass rod at the other. The glass rod is simply used to increase the length of the pedestal such that a short pedestal rod is compatible with the mechanical drive system. Unfortunately, there will always be some small degree of tilt between the collet and the pedestal rod, so the tip of the pedestal may move a few tens of microns or more during the growth of a long fiber. This small horizontal movement of the pedestal tip as the pedestal moves upward affects the molten zone as the edges of the beam fall off of the pedestal. Currently, small adjustments in the position of the parabolic focusing mirror are made manually during fiber growth to ensure that the focus always aligns perfectly with the center of the pedestal. These manual adjustments are easy to make using feedback from the video system. When a minor misalignment occurs, the incandescent laser spot is observed to change shape as shown in **Figure 7**. The visible changes present concurrent with misalignment will allow the implementation of an automated focal-spot alignment algorithm. To accomplish this, it will be necessary to replace the mechanical stage-micrometers on the focusing mirror mount with stepper-motor driven actuators. The existing video processing software would be augmented to include measurements of the interface at the bottom of the molten zone. The shape of the bottom interface would then serve as feedback data to adjust the mirror position. We also note that overall incandescence levels can be used to perform such an adjustment, since the brightest incandescence occurs when the laser is well-focused on the pedestal. This modification will likely be performed in the near future as funds permit.

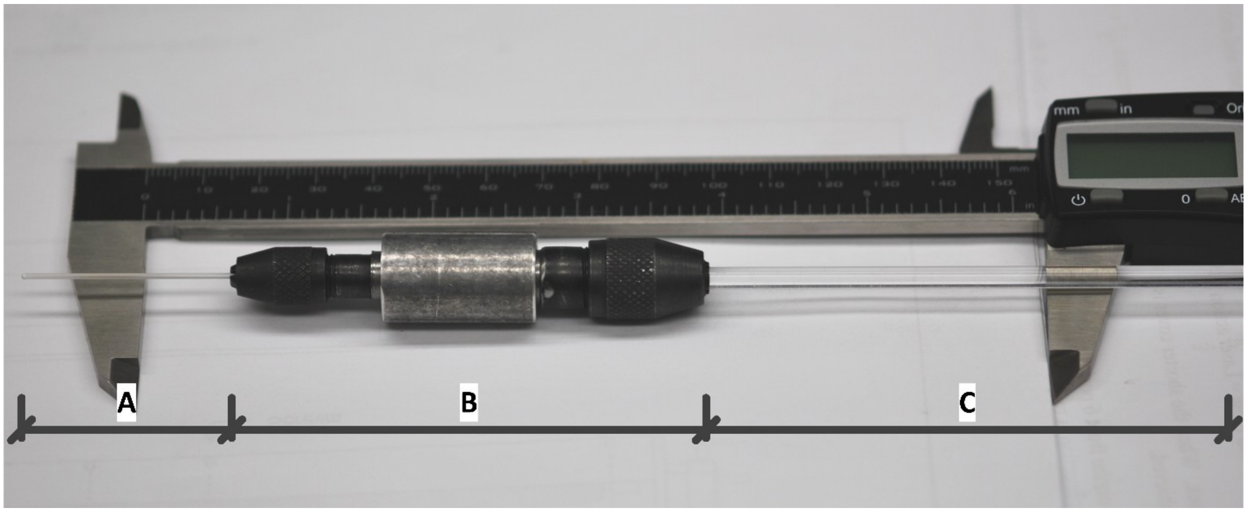

Figure 6. Pedestal extension device. A: 1 mm Sapphire pedestal, B: Double-ended collet fixture, C: 1/8" O.D. Fused silica extension rod

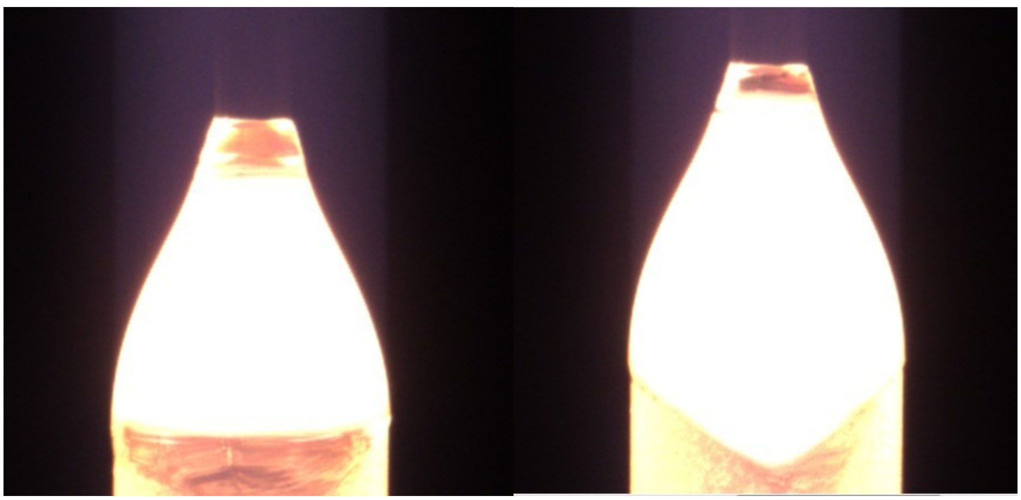

Figure 7. Aligned (left) and misaligned (right) laser focal spot

The last modification suggested here involves further improvement to the operating software implementation. The video measurement system appears to work rather well, and no modification is required for the camera and lens assembly. Video frames are provided at an adequate rate and with adequate resolution and contrast. Additional backlighting is necessary for the measurement of cold, grown fibers. A fiber backlight compatible with the telecentric lens system would be appropriate. After any modification to the drive system, it will be necessary to update the "recipe" and PID constants for each fiber or diameter ratio. Currently, several additional automation routines could aid future operators. The present method for introduction of a seed crystal involves manual centering and lowering of the seed crystal using the manual motor controls. An automated lowering algorithm could be implemented to eliminate this operator step, but is not necessary for basic operation. Some additional functionality could be added by permitting the operator to specify a varying desired diameter profile. This feature should be simple to implement given the present software architecture. Together, these modification will increase the system functionality and lower the learning curve for operators.

Other groups have suggested potential modification to increase the growth rate and fiber quality via control of the ambient growth atmosphere. Currently, there is a helium purge system present to fill the growth chamber with helium gas during operation. It has been shown that decreasing the pressure in the growth chamber is also useful for increasing growth rates. This is a somewhat difficult proposition, as numerous feedthroughs are required along with optical access for both the CO_2 laser beam and video system. Increasing the growth rate may eventually necessitate the addition of a vacuum chamber to reduce ambient pressures and further assure gas composition in the molten zone. That addition is likely to be one of the most expensive and complicated modifications possible. Currently, we plan to test the application of varying levels and compositions of purge gas at atmospheric pressure before the consideration of vacuum pressure operation. The addition of a vacuum chamber may inhibit the addition of other devices or systems for sensor research applications in the future.

5. CONCLUSIONS

A state-of-the-art LHPG system has been designed and constructed, and is now capable of producing high-quality single-crystal sapphire optical fibers. The existing research literature guided our design with an array of techniques chosen for their proven results and capabilities. Initial evaluation of the system's operation demonstrated reasonable stability and diameter control, although a number of improvements were noted which are likely to aid in fiber-quality. When considering the entire effort by a team of instrumentation designers, scientists, and engineers, the interdisciplinary nature of the complex LHPG system is apparent. A high-level of optical, mechanical, electrical, and software design was required to produce a functional system. Expertise in each of these areas was necessary, along with good communication between the team and external vendors and specialists, to complete the design and construction. Additionally, considerable consulting occurred with in-house safety professionals to ensure the safe operation of a high-powered laser system in a multi-use laboratory.

Now that the NETL LHPG system has been constructed, commissioned, and initially tested, the path is clear for an array of new experiments intended to reduce the cost and increase the functionality of high-temperature optical fiber sensors. One important effort being considered is the implementation of an in-process fiber cladding system, as an alternative to post-processing of the fiber to add a cladding. Systems of incorporating sensors directly on the grown-fiber via a number of deposition or processing techniques may also be explored. Presently, we have a significant amount of experience creating silica-fiber sensors using a number of techniques which can be extended to sapphire-fiber platforms. Methods such as sol-gel application-and-sinter, direct laser material modification, or others can be used for sensor fabrication in-process or after growth. The integration of the new LHPG system and our existing fiber-optic sensor fabrication methodologies, along with the growth of novel new materials will be of primary interest in our research going forward.

REFERENCES

[1] "Sapphire photonic crystal fibers", Pfeiffenberger, N., Pickrell, G., Kokal, K., Wang, A., Opt. Eng. 49(9), 090501 (September 02, 2010). doi:10.1117/1.3483908

[2] Jiang, H., Cao, Z. Yang, R., Xiao, H., Dong, J., "Synthesis and characterization of spinel MgAl2O4 thin film as sapphire optical fiber cladding for high temperature applications", Thin Solid Films, Volume 539, 31 July 2013, pp. 81–87

[3] "Single-crystal sapphire-based optical high-temperature sensor for harsh environments", Zhang, Y., Pickrell, G., Qi, B., Safaai-Jazi, A., Wang, A., Opt. Eng. 43(1), 157-164 (Jan 01, 2004), doi:10.1117/1.1629123

[4] "Single‒crystal fiber optical devices: A Nd:YAG fiber laser", Burrus, C., Stone, J., Appl. Phys. Lett. 26, 318, 1975, http://dx.doi.org/10.1063/1.88172

[5] "Optical properties of single-crystal sapphire fibers", Harrignton, J., Nubling, R., Applied Optics, Vol. 36, No. 24, 20 August 1997

[6] "Laser heated miniature pedestal growth apparatus for single-crystal optical fibers", Fejer, M., Nightingale, J., Byer, R., Rev. sci. inst., 55(11), Nov. 1984

[7] "Springer Handbook of Crystal Growth: Laser heated pedestal growth of oxide fibers", Marcello, R., Andreeta, R., Hernandes, A., pp. 393-432, ISBN: 978-3-540-74182-4

[8] Single crystal fiber optics, a review", Harrington, J., Solid State Lasers XXIII: Technology and Devices, Proc. of SPIE Vol. 8959, 895902-1, 2014, doi: 10.1117/12.2048212

[9] "The Reflaxicon, a New Reflective Optical Element, and Some Applications", Edmonds, W., Applied Optics, Vol. 12, No. 8, August 1973

[10] "Automatic diameter control system applied to the laser heated pedestal growth technique", Andreeta, M., Caraschi, L., Hernandes, A., Mat. Res., vol.6, no.1, 2003, http://dx.doi.org/10.1590/S1516-14392003000100019