OPTIMIZATION OF TM-DOPED PHOSPHOSILICATE GLASS FOR HIGH POWER FIBER LASERS

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Final Report

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1. SUMMARY

The goal of this project is searching for better thulium (Tm) host glass with an improved efficiency. We have been focusing on silica hosts with varying amount of phosphorus. Conventional Tm host has been heavily aluminum-doped silica. We have focused on three new key host compositions, 1) high P, low Al, 2), high P and high Al, and 3) low P and high Al. Corresponding passive fibers without Tm are also planned for each compositions to test background losses at ~2 μ m.

300W pump diode was acquired and the laser test bench was set up in the spring of 2015. Fibers with the first and the second compositions have been fabricated and tested for laser efficiency. Since the glass compositions are very different, unique fabrication process has been developed for each composition. The fabrication process for the third composition needed significant more efforts; it will take several more month to complete.

The efficiency of the first composition shows internal efficiency of ~80%, near quantum limited efficiency. This is very promising. Further work is planned to confirm this. The second composition worked poorly. This also needs to be further confirmed. We have also refined our characterization process for measuring background loss of passive fiber. The current results show the OH level in our fabrication process is adequate. There is evidence that a low background loss at ~2µm is key for high efficiency. This is still preliminary and needs to be studied in more details.

We have demonstrated some very promising results and started to provide critical understanding on how to improve Tm fiber lasers, but more studies are required to complete this optimization. The tasks and time schedule is summarized in table 1.

	1 st Q	2 nd Q	3 rd Q	4 th Q	5 th Q	6 th Q
Fabricate Tm-doped conventional fibers based on						
phosphosilicate glass						
Test laser efficiency and photo-darkening						
Optimize composition						
					\rightarrow	
Fabricate Tm-doped silica glass suitable for photonic bandgap fibers.						

Table 1.	Tasks	and	time	schedule
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List of tasks:

- 1. Fabricate Tm-doped small-core conventional fibers with varying compositions based on our base phosphosilicate glass composition
- 2. Test laser efficiency and photo-darkening
- 3. Develop an optimized composition of Tm-doped phosphosilicate glass for high efficiency and low photo-darkening
- 4. Fabricate Tm-doped silica core glass with an index uniformity of $< 5 \times 10^{-5}$ and an index matched to silica within 2×10^{-4} using a novel repeated stack-and-draw process.

2. INTRODUCTION

High power fiber lasers have been revolutionizing machining in manufacturing in the past decade. Increasingly, automotive industries are turning to the much improved throughput, flexibility, ease of maintenance and cleanness provided by fiber lasers at much lower operating costs. The next frontier is micro-machining at precision much beyond current mechanical tools with a focused laser beam and high throughput machining of much harder materials such as glass and ceramics, again way beyond the capability of current mechanical tools. Some of the key applications include drilling micron-size holes in fuel injection nozzles, fine surface texturing of jet fan blades for air flow controls, wafer dicing/processing, glass cutting for smart phones, etc. Laser particle acceleration is another emerging application, which can potentially shrink the km-long RF linear accelerator down to table size and make synchrotron sources much widely available for medical and scientific applications. Success of all these applications is critically dependent on further power scaling of fiber lasers.

High power single-frequency, single-mode fiber lasers are critical for achieving high-energy lasers with power beyond 100kW via coherent power combining. Recently under a HEL-JTO funded program, 800W was achieved in a single-frequency ytterbium-doped fiber laser using a combination of transverse acoustic profile tailoring and inhomogeneous Brillouin line-width broadening by axial temperature and strain variations in a PCF [1]. Single-frequency power of >5kW would require SBS threshold to be raised by another factor of >6, in addition to mitigating mode instability.

A few years ago, with HEL-JTO funding, Q-Peak, working with Nufern, scaled the power of 2µmwavelength Tm-doped silica fiber lasers to ~1kW [2], the second fiber laser to do so after ytterbium fiber lasers. The key to this is the efficient cross-relaxation process which enables slope efficiency in excess of 50% when pumped at ~790nm.

	Mode area	SBS Gain	SBS suppression via inhomogeneous broadening	SBS suppression via line-width broadening	SRS Threshold	Nonlinear self-focus threshold
Wavelength dependence	λ^2	No λ dependence	λ	λ^2	λ	λ^2
Increase over ytterbium	~4	1	=λ ² ×λ=λ ³ =~8	=λ²×λ²=λ4=~16	=λ ² ×λ=λ ³ =~8	~4

	Table 2. Wavelength	dependence	of key facto	ors influencing	nonlinear	thresholds
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Wavelength dependence of various nonlinear thresholds is summarized in table 2. In addition to the benefits of eye-safer and better atmosphere transmission at ~2µm, effective mode area of single-mode fibers scales as λ^2 . Even though SBS gain does not change with λ , SBS suppression via inhomogeneous broadening scales as λ and via signal line-width broadening scales as λ^2 [3]. If we can take full advantage of the mode area scaling of λ^2 at 2µm, the SBS threshold, therefore, will scale as λ^3 for inhomogeneous broadening via axially varying temperature and strain, and as λ^4 for signal line-width broadening, i.e. a factor of 8 and 16 increase over ytterbium fiber lasers respectively. SRS threshold will scale as λ^3 , i.e. a factor of 8 increase over ytterbium fiber lasers. Nonlinear self-focus threshold scales as λ^2 , i.e. a factor of 4 increase over ytterbium fiber lasers. The fiber laser is, therefore, a strong candidate for >5kW single-frequency single-mode fiber lasers and power scaling of fiber lasers in general.

Mode instability, however, also needs to be mitigated at high powers. It is currently observed in both LMA below ~3kW and PCFs below 1kW. Some theories have been developed [4, 5], including one developed at Clemson [6]. These theories, however, are still to be tested experimentally. Recently, Clemson has demonstrated robust single-mode ytterbium-doped all-solid photonic bandgap fiber lasers with 50µm core (PBF) (see figure 1), which should provide much better higher-order-mode (HOM) suppression than both current LMA and PCF and, consequently, provide higher mode instability thresholds. *Transverse mode instability roughly scales with waveguide strength of an optical fiber, i.e. V value of the fiber. For fibers with the same effective mode area, mode instability is expected to be much higher for thulium fiber laser than ytterbium fiber lasers.*

To summarize, thulium fiber lasers are much more superior in power scaling than ytterbium fiber lasers given the benefits of much higher nonlinear thresholds and transverse mode instability thresholds. The atmosphere transmission is also much better given that efficient operation above 2.1µm can be realized. This promise can only be realized if higher efficiency comparable to that of ytterbium fiber lasers can be achieved at high powers and advanced designs can be realized to exploit the potential for much large effective mode areas with much increased mode instability threshold. The key to >5kW single-frequency, single-mode fiber laser is, therefore, to fabricate robust single-mode large-mode-area Tm-doped fibers to fully exploit the SBS threshold increase provided by operating at ~2µm.



Figure 1) Robust single-mode 50µm-core ytterbium-doped all-solid photonic bandgap fibers demonstrated at Clemson [7].

The resulting optimized Tm-doped glass is critical for the realization of single-mode Tm-doped advanced optical fibers with much increased effective mode areas. A number of designs based on all-sold photonic bandgap fibers and leakage channel fibers are investigated at Clemson. Photonic crystal fibers and other designs are studied at other universities. The resulting Tm-doped fibers can lead to one to two orders of magnitude improvement in further power scaling of single-frequency fiber lasers, and direct generation of optical pulses with up to 20MW peak powers and pulse energy of many tens of mJ. These fibers are also critical for the generation of ultrafast optical pulses with up to TW peak power and many kW of average powers through fiber chirped pulse amplification (FCPA) scheme.

The resulting ultrafast high power fiber lasers have many applications. One major area of applications is micro-machining, i.e. machining at sub-micrometer precision and in hard materials such as glass, ceramic and crystal, at high throughput, which far exceeds the capability of current technologies. It is already revolutionizing many industries, e.g. drilling micron-size holes in fuel injection nozzles, surface sub-micron-dimension texturing for airflow control on jet turbine blades, precision glass panel cutting for smart phones, semiconductor wafer cutting and many others. These industries are already in the billion dollars and fast growing.

The high power ultrafast fiber lasers are also critical for laser particle accelerations, which can potentially reduce current RF accelerators from km to table-top scale, making it widely available for

scientific studies and medical treatments as synchrotron sources. It will significantly improve front end of lasers used in laser fusion. High power single-frequency fiber lasers are also critical for gravitational wave detections using extremely long interferometers.

Tm-doped high power fiber lasers at 2μ m are critical for eye-safer LIDAR and other optical surveillance applications. They also provide pumps for high power MWIR and LWIR sources, where there are many medical, spectroscopic and defense applications.

At Clemson, our research currently involves a large number of undergraduate, MSc and PhD students (~30 students in photonics areas). The program will also enhance our capability to provide students with research training and education in photonic, critical for our national lead in photonics industries and national defense.

3. METHODS, ASSUMPTIONS, AND PROCEDURES

The proposed work is to optimize glass host for high Tm-doping without clustering. Our plan is to use aluminum-doped phosphosilicate glass. We have done a lot of work with this host glass for ytterbium-doping [8]. It provides the highest rare earth doping levels with minimum clustering and is critical for minimal photo-darkening in ytterbium-doped fiber lasers. It can be made with the chemical vapor deposition system at Clemson, critical for the high purity required for high power operation. The glass is optimized by making a series of Tm-doped conventional fibers with varying levels of phosphorus, aluminum and thulium. These fibers will be tested for both lasing efficiency and photo-darkening using a 790nm pumping scheme. The cross relaxation process for 790nm pumping can be enhanced at high thulium doping levels. The increase of thulium doping level possibly up to 9wt% in the proposed aluminum-doped phosphosilicate glass is expect to provide an improved efficiency of >70%, comparing to the current ~50% at high powers [2].

Photo-darkening is believed to be associated with up-conversion of thulium ions due to clustering and absorption related to glass defects in the UV and visible parts of the spectrum. Phosphorus-doping is known is to minimize clustering in ytterbium fiber lasers and glass defects in silica glass in general [9]. Aluminum-doped phosphosilicate glass is, therefore, very promising for minimizing photo-darkening in thulium fiber lasers.

A large number of Tm-doped optical fibers with varying compositions will be fabricated during the course of this project. These fibers will be tested in a laser configuration pumped at ~790nm to evaluate their laser efficiency. Photo-darkening will also be characterized at various pumping levels to access loss increase over a wide range of wavelength. These results will be used in a feedback process to refine glass compositions to achieve the highest efficiency and an accepted level of photo-darkening. The ultimate objective of the project is to identify the optimum compositions of thulium-doped aluminum-doped phosphosilicate glass for incorporation into Tm-doped advanced optical fibers with much increased effective mode areas and mode instability threshold.

For each composition, a preform is fabricated first on a MCVD system. The preform is then drawn on a fiber drawing tower into optical fibers for evaluations. Clemson University has the only academic facility in the United States capable of fabricating optical fiber from a full complement of materials. The Optical Fiber Laboratory is comprised of an SG Controls MCVD lathe and Heathway Fiber Draw Tower along with a preform and fiber characterization laboratory.

3.1 Modified Chemical Vapor Deposition (MCVD) Lathe

The MCVD lathe was built by SG Control (figure 2 on the left). It has an extended bed with a motorized tailstock and double jaw chucks on both headstock and tailstock capable of securing glass preform stock up to 1.5 meters in length and 70 mm in diameter. Preform temperature is monitored with a tracking

pyrometer that provides feedback for flow to a half-round hydrogen/oxygen torch with a nitrogen curtain capability. The lathe is also equipped with an automatic tube diameter control and auto-soot extract, auxiliary hand torches and tube straightening fixtures. The entire apparatus is housed in a clean room enclosure with air flow control.

The lathe is interfaced through QNX software using a recipe-based control for all process variables. This interface allows for carriage speed, mass flow and tube temperature changes to be set or modified at any time during the process. Liquid precursors are delivered to one of four bubblers with individual oil bath control and isothermal heaters to maintain constant temperature under high-flow via a remote auto-fill system linked by Allen Bradley Programmable Logic Controllers (PLCs). Additional gases are routed through a gas drier/purifier system using electro-polished stainless steel tubing. Brooks digital mass flow controllers (MFC) with a high and low MFC control for each process gas are used for gas metering. The lathe is plumbed for N2, O2, H2, Cl2, He, SF6, SiF4, Freon, BCl3, SiCl4, GeCl4, POCl3, two additional spare gases and one spare liquid precursor.

All waste gases generated during preform fabrication are captured and sent to the pollution abatement system. This system, comprised of a ventri-particle collector and V/F scrubber system manufactured by Tri-Mer Corporation, eliminates solid particles, and neutralizes the waste stream. All captured solid particles are reclaimed for their germanium content. Additional items on the MCVD lathe are a Litton glass working lathe and a solution doping/etch cabinet. The glass working lathe allows for precision set-up of the handle, preform and exhaust tube assembly along with fire polishing. The cabinet set-up provides location to solution dope MCVD performs with a variety of rare earth solutions and to etch glass prior to fire polishing or fiber draw.



Figure 2) Clemson University's state-of-the-art MCVD and lathe (left) and fiber draw tower (right) facilities

3.2 Optical Fiber Draw Tower

The centerpiece of this laboratory is a 6.5 meter optical fiber draw tower equipped with three (3) interchangeable furnaces that cover temperature ranges from ambient to 2200 °C (figure 2 on the right). These furnaces include a high temperature graphite resistance furnace with a maximum temperature of 2200 °C used to draw silica materials. The high temperature furnace has an interchangeable graphite element package to accommodate preforms up to 50 mm in diameter. An inert argon atmosphere within the furnace protects the graphite form oxidizing during high temperature use.

Two intermediate temperature furnaces are used for soft glasses and polymer draws. The first unit is a single zone furnace with a maximum temperature of 1200 °C. This furnace can accommodate a maximum preform diameter of 50 mm. Dual gas controls are used for atmosphere and thermal draft

stability at top and bottom of furnace. The second intermediate temperature furnace has two independent heat zones, including a preheat and primary zone. This unit has a maximum temperature of 500 °C. Two independently controlled cooling zones for chilled water, above and below primary heat zones, are used to accurately control the temperature profile in the furnace. A dual gas delivery system provides atmosphere and draft control. This furnace has a narrow primary heat zone for tight thermal control. A maximum preform diameter of 20 mm can be drawn with this furnace. It is ideally suited for small exotic glass compositions and polymers.

The draw tower can handle preforms from < 5 to 50 mm. Line speeds up to 180 m/min are possible with forced cooling needed above ~ 80 m/min. Laminar HEPA filtered air flows across the draw line to maintain a particulate free environment. Dual diameters gauges, before and after coating, accurately monitor the fiber diameters from 30 to > 6000 μ m. The tower is also equipped with an automatic preform x-y adjustment unit. Two tractor units provide additional pulling capacity on the tower. A starter tractor assembly draws the fiber at a steady rate and consistent diameter for feeding the fiber into the solid die coating systems. A cane tractor assembly provides the capability of drawing rod or tubes of diameters from 500 to 6000 microns.

The draw tower is also outfitted with equipment to provide extensive fiber coating capability, including two coating stages for dual coating. Two solid coating heads have interchangeable dies based upon desired coating thickness, fiber size, and coating type. A single split coating die is available for more brittle glasses. Flexible tip silicon dies are used for specialty coating diameters or fiber types. Dual concentricity monitors provide information on the coating stability and evenness. Coatings can be cured with dual UV curing lamps or a single thermal curing furnace.

3.3 Photonic bandgap fiber fabrications

Tm-doped photonic bandgap fibers will be eventually fabricated at Clemson. We have been making photonic bandgap fibers through a well-established stack-and-draw process. Canes with desired geometries and compositions are fabricated first and then assembled on a stacking station (see figure 3). The stack is then inserted into a large tube and caned into preform rods on the drawing tower with a combinations of pressure and vacuum controls. The preform is then drawn into optical fibers for testing.



Figure 3) Stacking process.

4. RESULTS AND DISCUSSIONS

4.1 Characterization system

The 975 pump diode was ordered from DILAS in November 2014 and was delivered in March 2015. The quoted lead time was 12-14 weeks. It is a conductively cooled diode laser module operating at 795 ±3nm emitting 300W CW from a 200um/0.22NA 5m fiber. The chiller has an air/water heat exchanger capable of 900W of cooling and a water cooled power supply capable of 80A and 18V CW or pulsed with front panel controls. The cooling and driver was set up in May. The pump diode became operational in June (see figure 4).



Figure 4) 300W pump diode at 795nm and Tm fiber laser characterization system.

4.2 Fabrication of Tm-doped conventional fibers based on phosphosilicate glass

This is the main focus in the first year. Initial focus was on increasing phosphorus doping levels. This has been optimized in this period. One significant issue was that the fabrication process had to be significantly re-adjusted after each change. Further refinement is still needed. A summary of the fabricated Tm-doped preforms with the first target compositions (high P, low AI) is given in table 3. Tm doping level is settled at ~4wt% and P at ~12wt%.

Table 3. A summary of fabricated fibers

Sample	solution used	Al	Р	Thulium	Tm max	SEM AI:Tm ratio
LA04115 - 280	Th01.1	2.58	11.90	8.51	9.56	0.3:1
LA04315-350	Th01.2	2.51	12.37	8.37	9.35	0.27:1
LA05715 -240	Th02	2.79	12.16	9.32	10.97	0.30
LA05715 -290		1.80	10.15	6.19	7.97	0.29
LA05715 -360		1.22	10.50	3.86	4.35	0.32
LA05715 -390		1.10	10.23	3.92	4.54	0.28
LA06215-260	Th03	2.24	12.25	7.51	8.12	0.30
LA06215-390		1.50	10.64	5.11	5.71	0.29
LA06415-370	Th04	1.90	10.78	6.22	6.95	0.31
LA07215-290	Tm05.1	1.87	11.74	5.61	7.48	0.33
LA07215-390		1.61	12.04	5.39	5.85	0.30
LA07615-260	Tm05.2	1.80	11.71	5.36	5.80	0.34
LA07615-330		1.81	11.57	6.05	6.78	0.30
LA07815-270	Tm05.3	1.92	11.58	6.37	7.47	0.30
LA08315-280	Tm05.4	1.6	10.64	5.21	5.99	0.31
LA08915-240	Tm 06.1	1.77	12.22	6.15	6.82	0.29
LA09915-190	Tm 06.2	1.27	11.09	4.22	4.64	0.30
LA09915-280		0.95	11.41	2.76	3.53	0.34
LA09915-280-2	cleaned	0.95	11.21	2.54	3.36	0.37
LA09915-350		1.39	12.49	4.53	5.30	0.31
LA10115-160	Tm06.3	1.24	11.10	3.50	4.00	0.35
LA10115-310		1.76	12.74	5.18	5.90	0.34
LA12615_270	Tm06.5	1.36	10.77	4.3	6.57	0.32
LA17615-190	TmFreon01.12	0.17	13.33	0.73	0.92	0.23
LA17615-360		0.24	13.48	0.95	1.14	0.25
LA17815-190	TmFreon01.13	0.16	13.09	0.59	0.78	0.27
LA17815-380		0.21	12.56	1.06	1.31	0.20
LA18215-400	TmFreon01.16	0.22	13.51	1.57	1.83	0.14

4.3 Fiber compositions

Two fibers were drawn once the fabrication process was optimized for the first composition. One is Tmdoped double-clad fiber for optical tests (see figure 5) and the second is without Tm doping for characterization of background loss (see figure 6). Several more fibers were made later for refined measurements.



Figure 5) Composition of the Tm-doped double-clad fiber fabricated for optical test (the first composition).

Preforms and fibers with the second compositions (high P, high Al) have also been fabricated along with a fiber without Tm doping. The composition and refractive index of a typical preform with the second composition is shown in figure 7. The initial laser test result is poor. Focus have been moved elsewhere.

Further investigation may be required here at a later stage. The fabrication process for the third compositions (low P, high Al) have been recently established. Fibers without Tm doping have been fabricated for loss tests. More fibers with varying P are planned. The composition and refractive of a preform with the third composition is shown in figure 8. A summary of all tested fibers are given in table 4.



Figure 6) Composition (left) and refractive index (right) of the fiber with Tm doping for checking background loss (the first composition).





Figure 7) Composition and refractive index of a preform with the second composition.

Figure 8) Composition and refractive index of a preform with the third composition.

Table 4. A summary	of tested fibers
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	Prefrom	Fiber	solution	ABS 1400 nm	ABS 2000nm	w/w%P	w/w%Al	AI:P	w/w%Tm	avg RI in core	avg core width
										(cross 0.002)	(cross 0.002) mm
Lasing	LA12615	DE19615				12.5%	1.58%	0.13:1	5.19%	1.035E-02	3.93
	LA12815	DC13315	Base Al01	0.16	0.205	11.4%	1.66%	0.15:1	0.00%	1.067E-02	3.82
	LA22415	DA27115	TmFreon02.6	1.92	5.96 (1900nm)	7.2%	0.34%	0.05:1	2.26%	2.501E-02	1.44
	LA25315	DC26715	Alum.08.1	0.53	0.47	10.2%	4.73%	0.46:1	0.00%	3.082E-03	1.27
SEM avg 165+120	LA04816	DA05316	Alum 10.7	0.071	0.106	7.5%	9.45%	1.3:1	0.00%	7.894E-03	1.45

4.4 Laser tests

Several fibers from composition 1 (high P, low Al) and 2 (high P and high Al) have been fabricated into double-clad fibers with various geometries for tests with the diode pump at ~790nm. No lasing action was observed in many of these fibers. One difficulty is high thermal load at the pump launching end. Pump absorption has to be reduced by changing core/clad ratio in several cases.

The best result is from a fiber with the first composition (high P, low Al). A cut-back measurement was conducted and the result is shown in figure 9. The lasing wavelength moved towards shorter wavelength as expected as the laser operated more like a three-level system. The highest efficiency versus the launched pump power reached ~40%. The efficiency versus the absorbed pump power went beyond 80% for shorter fibers. This is beyond quantum limit and is not possible. Further detailed tests are planned.



Figure 9) Efficiency and lasing wavelength of a fiber with the first composition.

4.5 Fiber loss characterizations

Loss measurement is also conducted on fibers without Tm doping to access the background loss near the laser wavelength. This may hold the key to further efficiency improvement, since it may be responsible for a fraction of the energy loss in a fiber laser.



Figure 10) Fiber background loss measurements.

The loss measured in the fabricated passive fibers without Tm doping is shown in figure 10. The OH related peak at ~2.2µm is not visible, indicating that OH is sufficient low in our fiber. The increasing loss towards longer wavelength due to phonon absorption is clearly seen. The loss level is ~0.1dB/m at ~2µm for the first composition. This can contribute towards 10-20% power loss in a fiber laser of several meter long. The loss peak at ~1.95µm is due to water absorption in air.

Loss for the second composition is also tested and shown in figure 8. The loss is significantly higher. No lasing action was observed in fibers with this composition. The high loss may be responsible. We have moved focused to more studies with the first and the third compositions.

Measured loss of the third composition looks promising. The fabrication efforts for active fiber is ongoing.

5. CONCLUSIONS

The first composition shows internal efficiency of ~80%, near quantum limited efficiency. This is very promising. Further work is planned to confirm this. The results show the OH level in our fabrication process is adequate. There is evidence that a low background loss at ~2 μ m is key for high efficiency. This is still preliminary and needs to be studied in more details. Overall, the program has demonstrated some very promising results and started to provide critical understanding on how to improve Tm fiber lasers, but more studies are required to complete this optimization.

6. LIST OF SYMBOLS, ABBREVIATIONS AND ACRONYMS

- Al: aluminum
- CW: continuous wave
- FCPA: fiber chirped pulse amplifier
- HEPA: high-efficiency particulate air
- HOM: higher order mode
- LIDAR: light detection and ranging
- LMA: large mode area
- LWIR: long wave infrared
- MCVD: modified chemical vapor deposition
- MFC: mass flow controller

- MWIR: mid wave infrared
- NA: numerical aperture
- OH: hydroxyls
- P: phosphorus
- PLC: programmable logic controller
- PBF: photonic bandgap fiber
- PCF: photonic crystal fiber
- SBS: stimulated Brillouin scattering
- SRS: stimulated Raman scattering
- Tm: thulium
- UV: ultra violet
- λ : Wavelength of operation

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