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RESEARCH ON SOLAR PUMPED LIQUID LASERS

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I. INTRODUCTION

The goal of this research effort is to develop a solar pumped liquid laser that can be scaled up to high power (10Mw CW) for space applications. Liquid lasers have the inherent advantage over gases in that they provide much higher lasant densities and thus high power densities. Liquids also have inherent advantages over solids in that they have much higher damage thresholds and are much cheaper to produce for large scale applications.

Among the liquid laser media that are potential candidates for solar pumping, the POCl₃:Nd³⁺:ZrCl₄ liquid has been chosen for its high intrinsic efficiency as well as its relatively good stability against decomposition due to protic contamination. The research effort thus far has been devoted towards the development and testing of the laser liquid and the development of a large solar concentrator to pump the laser.

The procedure to manufacture the laser liquid must include diagnostic tests of the solvent purity (from protic contamination) at various stages in the production process. In addition the final solution must be tested for lasant density and impurity concentration to insure good quality control.

All diagnostic procedures involve absorption scans of the solution. To check for protic contamination, an absorption scan of the solution is required in the infrared from 2.5 to 4 microns. An absorption scan in the visible and near IR region of the spectrum (500-900 nm) is required to determine the Nd^{3+} ion concentration in solution. The details of these procedures are described in the next section.

The lasing characteristics of the liquid are studied using xenon flashlamps to simulate the solar irradiance provided by the solar concentrator. Important parameters such as the minimum required pumping power and the maximum pulse width are required to design an optimum solar pumped laser cavity.

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One of the most significant accomplishments of this research effort has been the development of an inexpensive solar concentrator. It is capable of a concentration factor of better than 1000 suns and at the same time deliver a kilowatt or more at the laser cavity. The construction method used to build this facility as well as its performance characteristics are described in detail in Section V.

II. PREPARATION OF THE LIQUID LASER MEDIUM

A. General Concept

The objective of this project element has been to prepare an aprotic solution containing $0.3\underline{M} \ Nd^{3+}$ ion suitable for lasing. It is necessary that the solution be aprotic to avoid non-radiative quenching of the excited state. We chose the system which results from dissolving neodymium trifluoroacetate in phosphorus oxychloride (POCl₃) with the aid of zirconium tetrachloride (ZrCl₄). The choice of this solution in preference to other similar solutions was based on the relative stability of this solution, both under operating conditions and toward minor accidental water contamination. Phosphorus oxychloride was the preferred solvent because it is less corrosive and less toxic than selenium oxychloride (SeOCl₂). The system employing POCl₃ with ZrCl₄ as the Lewis acid is stable to UV radiation and, therefore, does not photodegrade. It has the additional advantage that ZrCl₄ reacts with water to produce a solid (ZrOCl₂) and a gas (HCl), thereby removing protic contamination from the solution.

B. Previous Difficulties

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We believe that our initial difficulties in affecting solution--we initially obtained large amounts of precipitate--were due to a combination of factors. These included "wet" POCla (it still contained in excess of 75 ppm's of protic species), wet ZrCl4, and handling techniques which were not sufficiently rigorous to maintain anhydrous conditions. These were corrected by treating the POC13 with lithium metal, purchasing a new supply of ZrCl4, and designing a new distillation apparatus which incorporated the use of syringe techniques to make all transfers. We had been reluctant to make the first of these changes--the use of lithium to decontaminate the POC13 of protic species-because drying POC13 with other alkali metals (e.g., Na or K) is reported to frequently result in an explosion. However, it was pointed out to us that the use of lithium was absolutely necessary to get the solvent sufficiently free of protic contamination. The other improvements were necessary to keep the solution free of protic contamination. What follows is a detailed procedure--including diagrams of our glassware--which may be used to produce the required lasing solution.

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C. Reagents

The following reagents were purchased and used as received:

neodymium oxide, Nd₂O₃ (99.999%, Alfa Products),

trifluoroacetic acid, CF₃COOH (TFA) (reagent grade, Fisher Scientific Co.), and

zirconium tetrachloride, ZrC14 (99.9%, Research Organic /Inorganic Chemical Corp.).

The phosphorus oxychloride, POCl₃ (reagent grade, Fisher Scientific Co.). was treated as described in a following section.

Lithium metal, Li (99.9%, Alfa Products), was obtained as shot, packed under argon with a coating of mineral oil on it. The mineral oil was removed by washing the metal with hexane (or other low boiling hydrocarbon) and collecting the lithium on a filter. The metal was then air dried (quickly, to avoid nitride formation) and transferred to a vacuum dessicator where the last traces of protic contamination (i.e., the hexane) were removed. The use of lithium dictates the use of argon for the inert atmosphere in subsequent distillations. The argon gas, Ar (99.998%, Airco) was always dried by passing it through a column of indicating Drierite (W. A. Hammond Drierite Co.) and Aquasorb (Mallinckrodt) before being used.

Anhydrous neodymium trifluoroacetate, $[Nd(CF_3COO)_3]$, was made by very <u>SLOWLY</u> adding 170 ml of trifluoroacetic acid to a mixture of ~50 g (accurately weighted) of Nd₂O₃ and 100 ml of water. The mixture was stirred then heated until all of the Nd₂O₃ dissolved. Water and excess TFA were then removed by evaporation, leaving a dark lavender solid, hydrated Nd(TFA)₃. The solid was then ground up and transferred to a vacuum oven to be dried to constant weight. This took several weeks with the oven operating at $\sim 60^{\circ}$ C. It was necessary to remove the solid from the oven periodically to regrind it and to expose a fresh surface. The theoretical yield (based on exactly 50 grams of Nd₂O₃) is 143.62 g. However, some loss does occur during the many transfers of the solid. The final product, anhydrous neodymium trifluoroacetate, is a pale blue-lavender colored powder.

D. Glassware

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Much of the glassware was fabricated to our specifications. Greasing of stopcocks and joints was avoided by the use of Teflon stopcocks throughout and Clear Seal (Wheaton Scientific) joints wherever possible. Teflon tape was used on all other joints to insure a tight fit. (Fluorinated greases should be avoided since they tend to absorb small amounts of water.) All glassware was routinely cleaned in chromic acid, rinsed, and oven dried at 120°C. In order to avoid contamination from atmospheric moisture, the glassware was removed from the oven immediately before it was used and assembled in our hood while still warm. Care must be taken to only loosely screw on the Teflon stockcocks initially, thus preventing the glass from cracking as it contracts. Only after the glass has cooled should they be tightened completely. The various distillation setups were each wrapped with glass wool to aid in maintaining uniform and constant temperature over the course of the distillation. The connections between glass pieces were made with Teflon -lined polyethylene tubing (e.g., between the still and the bubbler). We used two mineral oil bubblers, each with a check valve. Each opening for syringe entry was

capped with a 14 mm rubber septum.

E. Preliminary Treatment of the Solvent

Two consecutive simple distillations of the POCl₃ were performed under an atmosphere of argon. Only minimal precautions were taken at this stage to insure dryness. Each time only the middle fraction, boiling range $107.5^{\circ}-108.0^{\circ}$ C, was collected. (lit. bp = 107.7° C²). This was done to remove HCl, most of the water, and some of the higher boiling impurities. It is important to do this before adding the lithium metal to minimize the reaction between the lithium and the more reactive protic species. If the distillate was <u>completely</u> colorless, it was transferred to the final distillation apparatus (Fig. 1) and the lithium metal was added. (<u>CAUTION</u>: In one instance, when the POCl₃ was slightly yellowish, an explosion occurred after the lithium had been added. We speculate that this may have been caused by a volatile impurity which catalyzed a reaction between the lithium and the POCl₃.) From this point on, all operations were conducted under an atmosphere of argon.

F. Preparation of the Pure Solvent

The POCl3/Li mixture was then refluxed for 24 hours. This allowed the solvent to remove traces of water from the walls by "washing" the glassware. When the POCl3 returned to the distillation flask, the lithium reacted with any water present and also any protic species which had been formed by reaction with water, further drying the apparatus. Phosphorus oxychloride then distilled into the collection vessel by closing





the 6 mm stopcock. After a first cut was removed through the 2 mm stopcock, the pure, dry POCl₃ was collected. (Since our condensing column was too close to the thermometer in this apparatus, exact temperature readings were <u>not</u> used as a guide in distilling pure solvent.)

G. Preparation of the Lasing Liquid

The apparatus as seen in Fig. 2, connection scheme (1) was taken into a glove bag. Anhydrous neodymium trifluoroacetate (14.5 grams, 0.030 moles) and a magnetic stirbar were placed in the bottom of the reaction flask. Zirconium tetrachloride (10.5 grams, 0.045 moles) was placed in the solid addition funnel. The connection to the argon bubbler was protected with a rubber bulb and the flask was removed from the glove bag to the hood where a second argon bubbler was connected. Using a double-tipped needle, opening (A) (Fig. 1) was connected to opening (B) (Fig. 2). Gas pressure was used to transfer 100 ml of POC13 to the reaction flask. The mixture was stirred and some solid remained until the ZrCl4 was added by rotating the solid addition funnel. In some cases, it may have been necessary to allow the solution to flow into the addition funnel to get all of the ZrCl4 out of it. The order of reaction, mixing POCl3 with Nd(TFA)3 and then adding ZrCl4 is contrary to all published accounts of the preparation, but it is in accord with our direct communications with one of the original workers on the project.

When it appeared as if all of the solid had dissolved, the filtering funnel and the final distillation/storage flask were attached (see Fig. 2, connection scheme (2). The apparatus was then tipped to allow for





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filtering and the solution was collected in the "final distillation" flask. This flask was then attached to a simple distillation set up and the solution was distilled to one-half volume (50 ml) to remove volatile impurities that have accumulated over the many steps. The solution was then reconstituted to 100 ml by adding fresh POC13, using a doubletipped needle as before. The solution was stored in this flask.

H. Handling of the Laser Liquid

As shown in Fig. 2, the storage flask is sealed by two Teflon^R stopcocks, yet there is still access to the solution--without exposing it to air--through a rubber septum. We believe that this is sufficient protection for prolonged storage of the solution. However, we have not attempted to store a solution in this flask for longer than three months. Therefore, prolonged storage in this manner has not been fully tested.

As needed, a portion of the solution was removed with a 50 cc syringe and transferred to the laser cavity. The laser cavity which we used for all the data presented in this report was made by melting a Pyrex tube around the edges of both quartz windows. Since the quartz had not been melted, this procedure did not afford a complete seal and there was some leakage where the quartz and the Pyrex met. Apparently, the incomplete seal also allowed water to get into the cavity resulting in the formation of some precipitate. Over a period of time the viscosity and turbidity of the solution increased significantly, preventing the successful alignment of the laser cavity and mirrors. We believe that the presence of water was also responsible for a pressure build up, (possibly HCl gas)

within the cavity. The increased pressure only became a problem while opening the laser cavity. We believe that we have since corrected both of these problems by changing to an all Pyrex cavity having windows which are fused onto the main body of the cavity.

I. Spectral Properties--Vibrational

The $4000 \rightarrow 2500 \text{ cm}^{-1}$ range of the infrared region has always been our way of checking on the dryness of the POC13. We used a single 20 mm cell with supracil windows and a Perkin-Elmer 283 spectrometer to record all IR spectra shown in this report. However, a comparison between our IR spectra and other published spectra is difficult at best. The difficulty is the result of our use of cells with supracil windows and also other workers using cell path lengths which are either too short for our needs or unspecified in their publications.

The problem with supracil windows is that they absorb in the range $3800 \rightarrow 3500 \text{ cm}^{-1}$ with a maximum at ~3680 cm⁻¹ (see Fig. 3, "Blank"). The absorption can be accounted for and, therefore, this is only a minor complication. We have since corrected this problem by obtaining a matching pair of Beckman 20 mm cells with Near IR Silica windows. Each of these cells is transparent down to 2900 cm⁻¹ and with the other empty cell in the reterence beam the range can be extended down to 2500 cm⁻¹.

Some of the reported IR spectra of POC13 use much shorter path lengths than 20 mm. As a result, the absorptions that we need to examine for ppm amounts are undetected by them. This leaves us with only one source with





which to compare, the reports and papers from the GTE group. Even this source does not always state a path length so that accurate comparisons cannot be made.

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Previous reports of the IR spectrum of POC1₃ show the 3860 cm⁻¹ peak poorly defined, as little more than a distortion in the baseline.² It is clear from our results that this peak is real (see Fig. 4), although it is not certain whether or not this is an impurity or the inability of their spectrometer to resolve the peak. However, we believe this peak is characteristic of POC1₃ and is the second overtone of the P=0 peak reported at 1290 cm-1.

Before we began using our special glassware (Fig. 1), distillation of POC13 resulted in only a minimal decrease in the content of various partially hydrated species of $POC1_3$ --species like $PO(OH)C1_2$. However, when we distilled from lithium with the special glassware, the residual protic species were reduced substantially (see Fig. 4) and the IR of the final laser solution (Fig. 5) compares favorably with the reported IR.³ Compared to the GTE group, our spectrumis flatter (except for some small peaks) in the region above 2900 cm⁻¹ although it does drop off more sharply below 2800 cm⁻¹. However, once again it is difficult to interpret the significance of the rate of drop off since the path length is not reported.





Figure 5: IR of the Final Laser Solution

J. Spectral Properties - Electronic

Absorption spectra of the laser liquid can be used to determine if a sufficient concentration of the Nd^{3+} ion has been obtained in the solution. There are 5 absorption bands of the Nd^{3+} ion in the spectral region between 500 and 900 nm. As can be seen in Fig. 6, the absorption bands are present in sufficient intensity. The results shown indicate that the solution contains approximately a 0.3 molar concentration of Nd^{3+} ion. The spectra is obtained using a Tracor Northern (TN-1710) Optical Multichannel Analyzer attached to a 1/4 meter Jobin-Yvon grating spectrometer. The light source used was a calibrated tungsten ribbon lamp. The absorption cells used were Beckman 20 mm cells with Near IR silica windows.

III. MEASUREMENT OF SMALL SIGNAL GAIN AT 1.05 μ m

To measure small signal gain, the following procedure was used. The liquid is transferred to a quartz cell and placed in a flashlamp housing which contains two EGG FX-81-C-8 flashtubes. The flashtubes are energized by 7 capacitors (200 Joules each) in parallel. They are triggered by a pulsed 30 kV high voltage source. The flashlamp housing is placed on an optical bench in line with a 500 mW CW Nd³⁺:YAG laser. The YAG probe beam passes through the liquid cell and is detected by a PIN photo diode. A 1.05 μ m interference filter is placed in front of the photo diode for discrimination against other wavelength light. (See Fig. 7). The gain measurements are made as follows:

1. The flashlamps are fired with the probe beam off. The fluorescence intensity of the Nd^{3+} is measured. (1.06 μ m only).



Figure 6: Absorbtion Band of Neodymium



2. The same procedure is repeated with the probe beam on. The YAG probe beam produces an intensity output of about 10 volts in absence of flashlamp pumping. During the gain shots the intensity of the output beam is increased to about 24 volts, thus indicating a gain of about 2.4 (See Fig. 8a). Note that in 8b the gain is much lower and the pulse actually returns below the baseline. In this case, the liquid was still warm from the previous shot and gain was produced only during the pump pulse (50 μ sFWHM) and afterwards the liquid began to absorb the probe beam.

In the first shot - using a cool liquid - the gain lasts much longer, about 200 μ s as can be seen in Fig. 8. This compares to the upper state lifetime of the Nd³⁺ of about 330 μ sec. Therefore, the liquid produces gain until the upper state has been drained. For time comparison, Fig. 8 also shows the flashlamp pump pulse taken perpendicular to the gain axis (all wavelengths). The flashlamp emission indeed ceases before the gain goes to zero.

IV. LASING OF THE POC13:Nd³⁺:ZrC14 LIQUID BY XENON FLASHLAMP PUMPING

The experimental set-up for the lasing attempts was similar to the gain measurement set-up with the exception of the probe laser and the addition of cavity mirrors as shown in Fig. 9. The energy storage capacitor (7 25μ f capacitors @ 4kV) was connected to two EGG FX-81-C-8 Xe Flashlamps in parallel. They are fired by a 30 kV trigger pulse. The laser cavity consists of a 30 ml volume of the liquid 12" (30.5 cm) cavity length in a hemispherical cavity consisting of a flat 1" (2.54 cm) dia. 99.9% reflecting mirror and 2 m radius 60% reflecting output coupler. The laser output pulse



gain (cold liquid)

a) Upper Trace:
gain profile
50 µs/cm time base
5V/cm
(probe input 5V)

Lower Trace: Flashlamp Output



gain (hot liquid)

Figure 8. Gain Measurement

b) Upper Trace:
 gain profile
 50µs/cm time base
 2V/cm
 (probe input 5V)

Lower Trace: Flashlamp Output



is reflected off of a 99.9% reflecting mirror (at 1.06 μ m) blazed at 45° and focused (quartz lens) on a PIN Photodiode behind a 1.05 μ m interference filter. The flashlamp output is also monitored by a PIN DIODE. Both diode outputs are monitored by a 555 Tektronics Oscilloscope.

Fig. 10 shows the laser and flashlamp output. The time base is 20 μ s/cm. As shown the laser output is delayed from the onset of flashlamp excitation by about 20 μ s and has the characteristic ringing of high gain lasing systems. The energy stored in the capacitors during this shot was 100J and the estimated laser output energy is about 2J.

V. FABRICATION TECHNIQUE FOR LARGE FOCAL LENGTH PARABOLIC MIRRORS

To achieve either CW or pulsed lasing in glass or liquid lasers using solar radiation, it is first necessary to concentrate the incoming radiation by a factor of better than a thousand and at the same time be able to deliver one kilowatt or better at the target. Although such concentration can readily be achieved with a high quality optical system using parabolic mirrors or lenses, the expense for large area intercept systems such as needed in the solar pumped laser area would be prohibitive. Accordingly, we have constructed a concentrator facility using a new and relatively inexpensive approach based on the concept of the rotational casting of mirrors. Although these do not have the optical quality of a ground optical glass system, they are sufficient for the present intended purpose. The centrifugal mirror construction technique is an outgrowth of earlier work^{45,6} on similar mirrors but extends these studies by not only considering very large mirrors but also incorporating new foam-fiberglass



Upper Trace: Laser output (-2J) 20 µs/cm

Lower Trace: Flashlamp output (~100J) 20µs/cm



Upper Trace: Laser Output (~1.4J) 20 µs/cm

Lower Trace: Flashlamp output (~80J) 20µs/cm

Figure 10. Lasing of the POCl₃ : Nd ³⁺ : ZrCl₄ liquid by Xenon flashlamp pumping

techniques ideal for achieving mirror rigidity and weight reduction.

After several attempts at constructing small parabolic mirrors using spinning techniques, we settled on the construction method illustrated in Fig. 11. It consists of first carving the front face of a polyurethane block measuring 8 x 8 ft (2.44 x 2.44m) and 3" (7.6 cm) thick into the shape of a parabolic surface having a predetermined focal length here taken as 44 ft (13.4m). We chose a square cross-section as opposed to the usual circular shape because it allows considerable construction simplifications. The parabolic contour was established with the aid of an aluminum template which was attached to a rigid beam and could be pushed into the foam thereby guiding the carving process and allowing a surface accuracy to better than 3 mm. The cut in the center for the present mirrors was 5-1/2 cm deep leaving 2 cm of foam thickness below. At the four corners of the foam block the thickness remained at 3" (7.6 cm). It should be noted that the maximum depth of cut into the foam required is directly proportional to the square of the side length and inversely as the first power of the focal length. The construction method is thus limited to large focal lengths for the large area mirrors considered here.

Once the parabolic shape had been carved, the entire foam surface including the sides and back, was encased in fiberglass impregnated with epoxy resin. Upon drying, this formed a very rigid structure of light weight. Next a rectangular box with back ribbing was constructed. Its dimensions were such that the foam sheet could just be inserted. Extra fiberglass and epoxy resin was used to produce a bond between these two units to form a single rigid structure.

Rectangular box with rib backing for mirror stability Square foam block with front surface cut into a parabolic shape. Entire surface is encased with fiberglass cloth to provide regidity. Parabolic epoxy layer cast onto the foam sheet by centrfugal methods. The front surface is coated with self-adhesive aluminum mylar strips.



The combination was next placed onto the rotating system shown in Fig. 12. This system consists of a heavy stationary table anchored into the floor together with a rotating vertical shaft held by bearings and driven by a variable speed electric motor. The frame-foam combination was dynamically balanced on top of the drive shaft and set into rotation at a constant angular velocity which was monitored electro-optically. The rotation rate could be kept constant to within one percent over periods as long as eight hours. The rotation period for the present mirrors was maintained at 10.4 sec. This period is consistent with the formation of a 44 ft (13.4m) focal length parabolic surface formed when pouring a liquid into the rotating parabolic dish.

The fabrication of the final parabolic mirror surface was accomplished by pouring liquid epoxy into the spinning dish and letting it harden. It was found that two pours of 3.2 mm (1/8") depth each produced the best results. Typical solidification times were five hours with the rotation being maintained for well over eight hours to insure sufficient hardening. The final hardened surface is estimated to be with one millimeter of a perfect parabola over the entire mirror surface with the exception of a few small localized indentations believed due to the presence of small air bubbles trapped between the fiberglass and the foam surface during fabrication. Vibrations of the liquid epoxy layer during rotation were negligiblebut some problems were encountered due to the presence of air currents above the rotating dish, which caused imperfections in the surface layer. These were reduced considerably when the room airconditioning duct was closed. For proper solidification the relative humidity had to be main-



Figure 12: Facility for the Centrifugal Casting of the Final Parabolic Surface

tained at less than 70%.

The last stage of the mirror fabrication process involved placing strips of self-adhesive aluminized acrylic film (3M-FEK244) onto the hardened epoxy surface. Since this surface had small curvature there was no difficulty in applying two foot (0.58 meter) wide strips of the film directly onto the surface without wrinkling problems. Two 64 square foot (5.95m²) mirrors were placed in the vertical plane orientation required for the present concentrator facility over six months ago and have shown no signs of deterioration. When not in use, they are covered with a nontransparent plastic sheet. Their final weight is about three hundred pounds (~140kg) each with the majority of the weight due to the 0.66 cm (1/4") thick parabolic epoxy layer. It was found that the construction of these mirrors was less time consuming than the fabrication of the corresponding heliostats. The heliostats had to be optically flat to better than two millimeters in order to function as light collectors for the parabolic mirrors located 70 ft. (21.3m) from them.

VI. SOLAR PUMPED LASING OF A Nd³⁺:YAG ROD

A CVI YAG MAX Laser was placed in the focal plane of the solar facility discussed in the previous section. The top half of the pump light reflector was removed, exposing the YAG rod and pump lamp to the incoming solar flux.

The incoming power was estimated (from the calorimetry experiment described in the semi-annual report) to be about 800W. The output power

(at 1.06 μ m) was measured (with a Coherent calorimeter power meter) to be 75 mW.

The laser efficiency is about half of what it should be due to various scattering losses. In order to improve the performance of the YAG rod, a much improved final concentrator and custom built cavity is being designed, not only to improve the YAG performance, but to test the cavity design for eventual use with the liquid laser medium.

VII. CONCLUSIONS

The results thus far obtained show promise for eventual success of the solar pumping of the liquid laser. The method for preparation of POCl3: Nd⁺³:ZrCl₄ is now well understood so that reliable laser solution can be routinely manufactured. Gain and lasing have been demonstrated under flashlamp excitation. Also, an inexpensive, reliable solar collector facility has been constructed and is now operational. Thus, it appears that the integration of the laser liquid with adequate collected sunlight will produce the first solar pumped liquid laser system.

The immediate goals of the next phase of this research effort are to define the minimum pumping power and maximum pump pulse width characteristic of the POCl₃:Nd³⁺:ZrCl₄ laser liquid using Xenon flashlamp pumping. Then an attempt will be made to lase the liquid in the solar test facility.

In addition, there will be a further investigation into the doping of chromium (Cr^{3+}) in the laser solution to enhance the laser efficiency.

It may be possible to produce a liquid chromium laser by substitution the Cr^{3+} ion for the Nd³⁺ ion. If this is possible, a more efficient turnable (720-780 nm) laser is possible.

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