

## **Laser and thermal performance of a new erbium doped phosphate laser glass**

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### **ABSTRACT**

New erbium doped glass base compositions and sensitizer ion concentrations have been investigated. Laser, spectral, and thermo-mechanical properties have been tested. This study has resulted in a new erbium doped phosphate laser glass that exhibits improved thermal shock resistance and superior laser performance.

### **1. INTRODUCTION**

Erbium laser glasses have attracted much attention due to their capability for emission of radiation at the "eye safe" wavelength of 1.54 microns.<sup>1,2,3</sup> The development of Erbium glass laser systems has renewed the requirements for additional research into improved laser glass properties and performance. Flashlamp pumped Erbium glass lasers are limited to relatively low repetition rates. This is due to the low thermal conductivity and poor thermal shock resistance of the laser glass<sup>4,5</sup>. This investigation has focused on the effects of both new dopant levels and variations in glass base composition on the overall laser glass performance in terms of high thermal shock resistance and laser efficiency. This report describes the effects of variations in trivalent Ytterbium and Chromium ions on laser efficiency and the improved physical properties of a new glass base.

### **2. EXPERIMENTAL**

A series of new phosphate based glasses were investigated in terms of the effect of the glasses composition upon the thermal expansion coefficient and the ability to accept rare earth ions. One new phosphate glass composition was selected as a control for dopant concentration studies. Different Yb<sub>2</sub>O<sub>3</sub> and Cr<sub>2</sub>O<sub>3</sub> concentrations were doped into the new glass base in order to investigate their effect on the spectral properties and relative laser efficiencies at 1.5 $\mu$ m. This study has resulted in a new Er<sup>3+</sup> laser glass composition with optimized active ion concentrations. The spectral properties, thermo-mechanical properties and laser characteristics were measured for this new glass.

The batch was melted in a platinum crucible in order to obtain high optical quality glass. Glasses with excessive Hydroxyl ion (OH<sup>-</sup>) concentration exhibit serious quenching of the Er<sup>3+</sup> fluorescence. The OH<sup>-</sup> content of the glasses were controlled via chemical and atmospheric methods and monitored by measuring the transmission at 2.2 $\mu$ m through an optically polished 1.0 cm thick sample. Cylindrical rods with a diameter of 5mm and a length of 76mm were fabricated to test the relative laser efficiency. The rod ends were polished flat and parallel and were left uncoated. Laser efficiency measurements were conducted using a Kigre FEM580K pump chamber and a K-300 flashlamp. The laser

resonator was 16cm long and consisted of a high reflector with a 10m radius of curvature and a flat output coupler with a reflectance of 85% at 1.54  $\mu\text{m}$ . The pump energy was calculated from the discharge capacitance and the initial voltage on the capacitor. The discharge pulse duration was 1.9ms. Deionized water was used as a coolant in the majority of the experiments. Kigre Perfluoropolyether coolant (EC-01) was also used for laser efficiency measurements with the newly developed glass.

The thermal expansion coefficient of the glass was measured in a dilatometer and the transformation temperature ( $T_g$ ) and the softening temperature ( $T_s$ ) were determined from the expansion curve. The thermal loading limit induced by flashlamp pumping was evaluated by minimum input power needed to fracture the laser rod. Five rods were used to measure the thermal loading limit for each glass composition and the average value is reported.

### 3. Results and Discussion

#### 3.1 Investigation of glass base composition

A family glass compositions based upon  $R_2O$ ,  $MO$ ,  $Al_2O_3$ ,  $B_2O_3$ ,  $Yb_2O_3$ , and  $P_2O_5$  ( $R = K, Na$ , and  $Li$ ;  $M = Mg, Ca, Sr, Ba, Zn$ , and  $Pb$ ) were investigated. The effect of these various glass compositions on the thermal expansion coefficient and the glasses ability to accept  $Yb_2O_3$  was studied. It is well known that the thermal expansion coefficient is a key parameter for influencing the thermal shock resistance of a given glass system. The lower the thermal expansion coefficient, the higher the thermal shock resistance. The thermal expansion coefficient of current commercial  $Er^{3+}$  doped phosphate laser glass QE-7S is  $114 \times 10^{-7} \text{C}^{-1}$  ( $20 - 40^\circ\text{C}$ ). A thermal expansion coefficient of less than  $90 \times 10^{-7} \text{C}^{-1}$  is desired in order to improve the thermal shock resistance by a factor of two. It is also well known that higher  $Yb_2O_3$  concentrations will typically yield higher laser efficiencies in  $Er^{3+}$  laser glasses. Unfortunately, the  $Yb_2O_3$  concentration is limited in most phosphate glasses due to devitrification. As the glasses  $Yb_2O_3$  concentration is increased, it becomes much more prone to crystallization. Doping levels of greater than 30 weight percent  $Yb_2O_3$  were attempted in order to test the ability of the glass base to accept high concentrations of this oxide. The thermal coefficient of optical path length, as expressed in the terms of  $dn/dt + (n-1)\alpha$ , where  $dn/dt$  is temperature coefficient of refractive index,  $n$  is refractive index at laser wavelength and  $\alpha$  is the thermal expansion coefficient, should be as low as possible to permit efficient lasing while operating at high average input power. The temperature coefficient of refractive index and optical path length were calculated for each melt<sup>6,7</sup>. One glass composition was selected according to measured and calculated results. This glass base exhibits excellent thermal shock resistance and acceptable optical athermal behavior.

**3.2 Investigation of  $Yb_2O_3$  concentration** One of the distinctive advantages of the selected glass base was that it accepts high  $Yb_2O_3$  concentrations. The effect of the  $Yb^{3+}$  ion concentration on laser efficiency was investigated by changing the  $Yb^{3+}$  content from  $1.5 \times 10^{21}$  to  $2.25 \times 10^{21}$  ions/cc. Figure 1 shows the laser test results for different laser rods with different  $Yb_2O_3$  concentrations. The  $Cr_2O_3$  concentration was held constant in all of the test melts. The pulse repetition rate of the flashlamp was 1 Hz and the coolant was deionized water. Figure 1 indicates that the best  $Yb^{3+}$  concentration for such a laser system is around  $1.8 \times 10^{21}$  ions/cc and higher concentrations cause a decrease in efficiency. An increase of the  $Yb^{3+}$  concentration results in an increasing absorption of pump energy and non-radiative energy transfer from  $Yb^{3+}$  to  $Er^{3+}$ , thus improving the  $Er^{3+}$  laser efficiency. On the other hand, excessive  $Yb^{3+}$  concentrations will cause an increase in the back transfer rate from  $Er^{3+}$  ( $4I_{11/2} - 4I_{15/2}$ ) to  $Yb^{3+}$  ( $^2F_{7/2} - ^2F_{5/2}$ ). In addition, high  $Yb^{3+}$  concentrations will lead to inhomogeneous pumping in 5mm diameter test rods.

The  $Yb_2O_3$  concentration in this new glass base may be increased to over  $3.0 \times 10^{21}$  ions/cc. This figure is two times the concentration of the current commercial  $Er^{3+}$  laser glass QE-7S. Such high  $Yb^{3+}$  concentrations may be

beneficial for diode pumped  $\text{Er}^{3+}$  glass lasers and/or small diameter or very thin gain medium configuration flashlamp pumped lasers. In a diode pumped Yb:Er glass laser, the pump intensity to reach threshold depends upon the absorption coefficient of  $\text{Yb}^{3+}$  when pumped around the 970nm spectral region. The direct absorption of  $\text{Er}^{3+}$  is negligible due to very low  $\text{Er}^{3+}$  concentration<sup>8</sup>. Therefore, a high  $\text{Yb}_2\text{O}_3$  concentration is desired for a diode pumped Yb:Er glass laser. Of course, the best  $\text{Yb}^{3+}$  concentration for a certain laser strongly depends on laser design and operating conditions. The new glass base provides a very flexible test bed to test and obtain the most suitable  $\text{Yb}^{3+}$  concentration for a given type of laser.

### 3.3 Investigation of $\text{Cr}_2\text{O}_3$ concentration

$\text{Yb}^{3+}$  sensitized  $\text{Er}^{3+}$  laser glass only absorbs a relatively small percentage of flashlamp pumping energy; therefore,  $\text{Cr}^{3+}$  was introduced as an auxiliary sensitizer in order to enhance the overall flashlamp pumping efficiency<sup>9</sup>.  $\text{Cr}^{3+}$  has two wide absorption bands centered at 460nm and 650nm. These bands are able to effectively transfer flashlamp energy to  $\text{Yb}^{3+}$  and  $\text{Er}^{3+}$ . The effects of  $\text{Cr}^{3+}$  concentration on laser output efficiency is shown in Figure 2. The slope efficiency was increased about 100% and the threshold was decreased about 20% by doping a suitable amount of  $\text{Cr}_2\text{O}_3$  into the glass. Figure 2 indicates that an optimum  $\text{Cr}^{3+}$  concentration is approximately  $1 \times 10^{19}$  ions/cc. A side effect of  $\text{Cr}_2\text{O}_3$  doping is the additional thermal storage which is induced into the glass laser rod. Although there is a good spectral overlap of  $\text{Cr}^{3+}$  fluorescence and  $\text{Yb}^{3+}$  absorption in most glasses, the quantum efficiency of energy transfer from  $\text{Cr}^{3+}$  to  $\text{Yb}^{3+}$  is not higher than 0.7 even at the highest  $\text{Yb}_2\text{O}_3$  concentration<sup>10</sup>. The thermal storage induced by doping  $\text{Cr}_2\text{O}_3$  causes a larger temperature elevation in laser glass rod. It is well established that fluorescence yield decreases with elevating temperatures in laser glasses<sup>5</sup>. In addition, the increase in temperature will add to thermal lensing; therefore, the  $\text{Cr}_2\text{O}_3$  concentration has to be optimized, especially in light of the fact that this new glass is intended to be used in high average output power applications.

**3.4 Properties of the new glass** The new  $\text{Er}^{3+}$  doped phosphate laser glass, designed QX/Er, was developed by doping optimized concentrations of various sensitizers into the selected glass base. Figure 3 shows the absorption spectra of this new glass from 350 to 1650nm. The spectra between 900nm and 1020nm was measured using a 1.8mm thick sample and the spectra between 350 - 900nm and 1020 - 1650 nm was measured using a 9.8mm thick sample. The major optical and physical properties of QX/Er are listed in Table 1. For comparison, the corresponding values for QE-7S commercial  $\text{Er}^{3+}$  doped phosphate laser glass are also listed in Table 1. Figure 4 shows the relationship between the laser output energy at 1.54 $\mu\text{m}$  and the input pump energy for QX/Er and QE-7S at 1 Hz and 0.2 Hz flashlamp pumping repetition rates. The test conditions were the same as described in Section 2. For each test, the laser was aligned at 65J input. The input energy was then decreased and increased to obtain various output energies. The slope efficiency for QX/Er, calculated between 50J and 80J input energy, was found to be 2.0% and 2.2% at 1 Hz and 0.2 Hz repetition rates, respectively. The values for QE-7S were 1.9% and 2.3% at 1 Hz and 0.2 Hz, respectively. Laser thresholds were obtained by extending the regression curve to zero output energy. The threshold was determined to be 35.5 J for QX/Er and 35J for QE-7S at 0.2 Hz. Figure 4 shows that QX/Er exhibits a smaller decrease in slope efficiency with an increase in repetition rate when compared to QE-7S. This advantage of QX/Er is explained by higher thermal conductivity and an optimized doping concentration designed for high repetition rate lasers. It should be noted that the test conditions were selected for reasons of convenience and were not optimal for specified laser performance. It is expected that the threshold and efficiency of these tested glasses will improve after laser optimization and the application of antireflection coatings to the faces of the test rods.

All of the laser efficiency measurements mentioned to this point were cooled with deionized water. Unfortunately, water absorbs some of the flashlamp light from 950 to 1100nm. This absorption obstructs the major  $\text{Yb}^{3+}$  absorption band. Figure 5 shows a comparison of laser characteristics for

OX/Er using deionized water versus perfluoropolyether as a coolant. Perfluoropolyether exhibits a high transmission from the UV to 2000nm. Figure 5 indicates that the laser performance improves significantly when perfluoropolyether is used as a laser coolant.

The thermal shock resistance of the glass is expressed as the thermal loading limit. This number is calculated from the maximum flashlamp input energy times the repetition rate for a given size rod. Such an expression for thermal shock resistance is much better suited to practical applications than terms based upon the maximum temperature difference to cause fracture in a given size glass article. The thermal loading limits for QX/Er and QE-7S are 360W and 130W respectively for 5mm diameter by 76mm length rod cooled with deionized water. The thermal shock resistance of QX/Er glass is close to three times that of the commercial QE-7S material, and thus allows for much higher repetition rate and average powers to be obtained. Figure 6 shows a comparison of the maximum recommended operating and breakage range of QX/Er and QE-7S glasses. The relationship between input power and output power for QY./Er at 2HZ repetition rate is also shown in Figure 6. This result was obtained in the standard laser system test station described in Section 2 using deionized water as coolant. More than 4 watts of average power and 1.3 KW of peak power at 1.54  $\mu\text{m}$  were obtained during preliminary testing. A significant improvement in the chemical durability of the new glass is also noted. The weight loss determined from a standard durability test on QX/Er is only one third of that of QE-7S and comparable to a silicate base laser glass. In addition, the QX/Er base allows for higher  $\text{Yb}_2\text{O}_3$  concentrations, which are often desired for laser pumping or flashlamp pumping Er:glass research.

#### 4. Conclusion

A new glass base has been developed which possesses a low thermal expansion coefficient, an acceptable optical athermal behavior, a high chemical durability, and an ability to accept very high  $\text{Yb}_2\text{O}_3$  concentrations. The effects of  $\text{Yb}_2\text{O}_3$  and  $\text{Cr}_2\text{O}_3$  concentration on laser efficiency were investigated. The best doping concentrations were found approximately  $1.8 \times 10^{21}$  and  $1 \times 10^{19}$  ions/cc, respectively. New  $\text{Er}^{3+}$  doped phosphate laser glass, QX/Er, exhibits high thermal shock resistance in combination with superior laser performance. The laser and thermo-mechanical properties of this new glass make it an excellent candidate material to meet the demanding requirements of medium and high repetition rate eye-safe lasers.

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**TABLE 1. PROPERTIES OF QX/ER AND QE-7S**

<b>PROPERTY</b>	<b>QX/ER QE-7S</b>	
CENTER LASING WAVELENGTH (nm)	1535	1535
STIMULATED EMISSION CROSS SECTION ( $\times 10^{-20}\text{cm}^2$ )	0.8	0.8
FLUORESCENCE LIFETIME (ms)	7.9	8.0
INDEX OF REFRACTION ( $n_d$ )	1.533	1.542
INDEX OF REFRACTION AT 1.535 $\mu\text{m}$	1.521	1.531
TEMPERATURE COEFFICIENT OF REFRACTIVE INDEX $dn/dt$ (20 - 40°C) ( $\times 10^{-7}, \text{C}^{-1}$ )	-10	-63
TRANSFORMATION TEMPERATURE (°C)	450	462
SOFTENING TEMPERATURE (-C)	485	
THERMAL EXPANSION COEFFICIENT ( $\times 10^{-7} \text{ }^\circ\text{C}^{-1}$ ) (20 – 40°C)	82	114
DENSITY (g/cc)	2.90	3.14
WATER DURABILITY (WEIGHTLOSS %) ( $\text{H}_2\text{O}$ , 100°C, 1Hr)	0.012	0.027