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Chemically Strengthened Er^3, Nd^3 Doped Phosphate Laser Glasses

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ABSTRACT

Significant enhancement of the thermal loading capability has been achieved with both Er^{3+} and Nd^{3+} doped inherently strong glasses by an ion-exchange chemical strengthening process. A free running laser with an average output power of 6.5 W and a Q-switched single mode laser with an energy of 5 mJ at a repetition rate of 15 Hz have been demonstrated at the 1.54 µm eye- safe wavelength with strengthened QX/Er glass. An average output power of 110 W at 1.05µm has been obtained employing a strengthened QX/Nd glass rod.

Keywords: Er^{3+} laser glass, Nd^{3+} laser glass, chemical strengthening, Er^{3+} aser, high average power, high repetition rate, eye safe laser

1. INTRODUCTION

Laser glasses are able to be cast into a variety of forms and sizes with excellent homogeneity, uniformly distributed doping ion concentrations, and relatively low cost. The major drawbacks are the low thermal conductivity and the low r-upture strength. The temperature gradient created in a laser glass element by a flash lamp pump source and surface cooling results in thermally induced stress. If the thermally induced stress exceeds the rupture strength of the glass, a failure of the laser glass element will occur. Thus laser glasses are typically limited to low thermal loading operations.

An improvement of thermal loading capability of laser glass could be achieved by increasing the inherent rupture strength of the glass or with special preparations such as tempering, ion-exchange strengthening or surface coatings. In order to increase the inherent rupture strength, a phosphate laser glass base exhibiting high strength and low thermal expansion coefficient as well as acceptable optical athermal behavior has been developed [1].

Cr^{3+}:Yb^{3+}:Er^{3+} and Nd^{3+} doped glasses, lasing at 1.54,um and 1.05 respectively, have been successfully demonstrated[2]. Laser experiments indicate both Er^{3+} and Nd^{3+} doped glasses exhibit superior laser performance. This paper describes our effort at further enhancement of the thermal loading capability of these two glasses by an ion-exchange chemical strengthening process. The chemical strengthening process and the laser performance of both strengthened glasses are presented.

2. ION-EXCHANGE CHEMICAL STRENGTHENING

2.1 Background

The thermal loading capability of active laser elements depends on the thermal shock resistance and the fraction of the flashlamp power dissipated as heat in these elements. The thermal shock resistance in glasses is expressed as[3]:

$$\mathbf{TSR= \sigma f K(l-\mu)/\alpha . E} \qquad (1)$$

where σf, K, μ, α, E are the fracture stress, the thermal conductivity, Poisson ratio, coefficient of thermal expansion and Young's modulus, respectively. The parameters in equation (1) are intrinsic properties with the exception of the fracture stress, which depends on the intrinsic strength, surface preparation quality, and residual stress. Since laser glass always falls in tension and fractures generally originate at the surface of the glass, a compressive surface residual stress is used to increase the thermal shock resistance. The best known method of introducing a compressive residual stress is tempering wherein the glass is cooled at a rapid rate down through its strain point causing the surface layer solidify before the interior of the glass. The subsequent cooling of the interior of the glass establishes a compressive stress. This is not an attractive method to apply to optical quality glass. One alternative approach is ion-exchange chemical strengthening. In this method larger ions in a molten-salt bath diffuse into the glass and occupy the vacancies of smaller ions which have migrated from the glass surface into the salt bath[4] . Both QX/Er and QX/Nd glasses are comprised of a large mole percentage of Li_2O. When these glasses are exposed to KNO_3 and $NaNO_3$ molten salt, the larger Na^+ and K^+ ions in the salt will replace the smaller Li^+ ion in the glass. This strengthening method may be applied to a variety of configurations without loss of optical quality of the active element. This is due to the fact that the surface compressive layer created is extremely thin and the stress distribution of the interior is approximately uniform.

2.2 Ion-exchange strengthening experiments

The glass strengthening process was investigated by treating fine ground cylindrical rods and varying the exposure time and bath temperature based on previous development experience with Q89 strengthenable phosphate laser glass. Two methods were employed to evaluate the strengthening. One method was to determine the thermal shock resistance. Treated cylinders with dimensions of 5 mm diameter by 20 mm long were put into an oven with a prescribed temperature and maintained at that temperature for one hour, and then were plunged into room temperature water. The temperature difference between the oven and the water required to produce fracture was taken as the value of thermal shock resistance. The test was repeated on the same cylinder, increasing the temperature with each successive cycle until the sample was fractured. The thermal shock resistance was determined from the results of six cylinders with an uncertainty for each data of + /- 10^oC.

The other method was to determine the maximum thermal loading capability of treated cylindrical rods with a configuration of 5 mm diameter by 76 mm long pumped with a continuous Krypton arc lamp in a Kigre FEM580K cavity. The input power of the lamp was increased until the test rod was fractured using a Helium Neon laser as a probe to observe the status of the rod.

2.3. Ion-exchange strengthening results and discussions

The ion-exchange mechanism is normally expressed as a diffusion process. The concentration distribution of the larger ions diffused into the glass is determined by Fick's second law. Generally, improvement of the thermal shock resistance is linear and proportional to the square root of the treatment time in the salt bath[5]. Figures 1 and 2 illustrate the minimum and maximum thermal shock resistance of six test rods vs. the square root of the treatment time in the salt bath for QX/Er and QX/Nd glasses, respectively. The relationship for both glasses is close to the generally believed linear relationship. Since QX/Er glass contains a certain amount of K_2O, the improvement slope of the thermal shock resistance for QX/Er is smaller than that of QX/Nd glass. The thermal shock resistance is doubled for QX/Er and tripled for QX/Nd by treating glasses in the salt bath for 24 hours. The compressive residual stress derived from equation (1) indicates that it is greater than the intrinsic strength after glass rods were treated in the salt bath 24 hours for QX/Er glass and 5 hours for QX/Nd glass. It should be pointed out that a number of parameters would impact the net compressive stress. The most complex parameter is the salt bath temperature, especially for phosphate based glasses. The salt bath temperature has to be high enough to result in efficient ion-exchange process while remaining low enough to prevent stress relaxation due to glass structural rearrangement at the ion-exchanged surface, and corrosion of the glass surface.

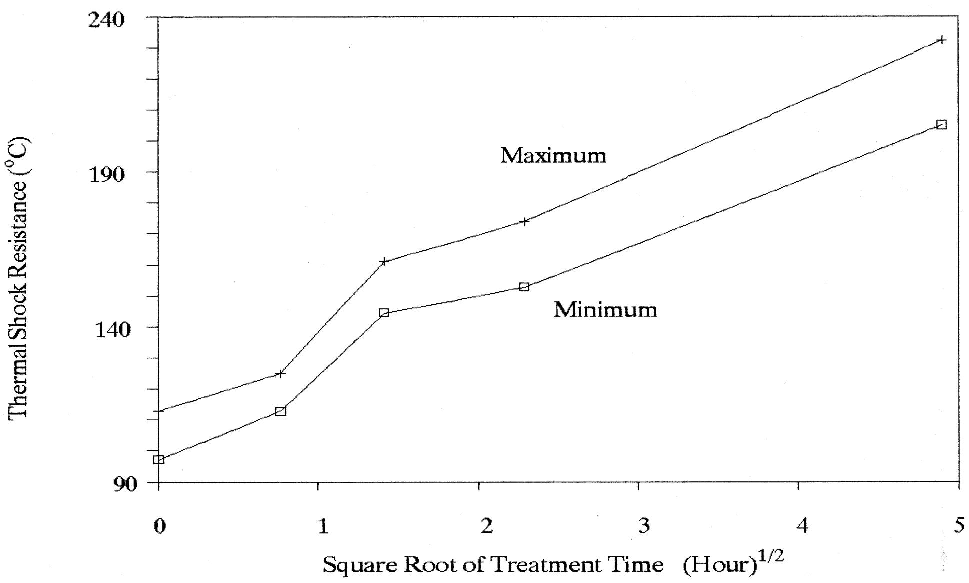

Figure 1. Thermal shock resistance of QX/Er glass vs. square root of the treatment time in the salt bath

For a laser glass rod pumped with a continuous lamp, the temperature difference between the rod surface and the center may be expressed by equation (2) with the assumption of uniform internal heat generation and extreme cooling solely along the cylindrical surface[6].

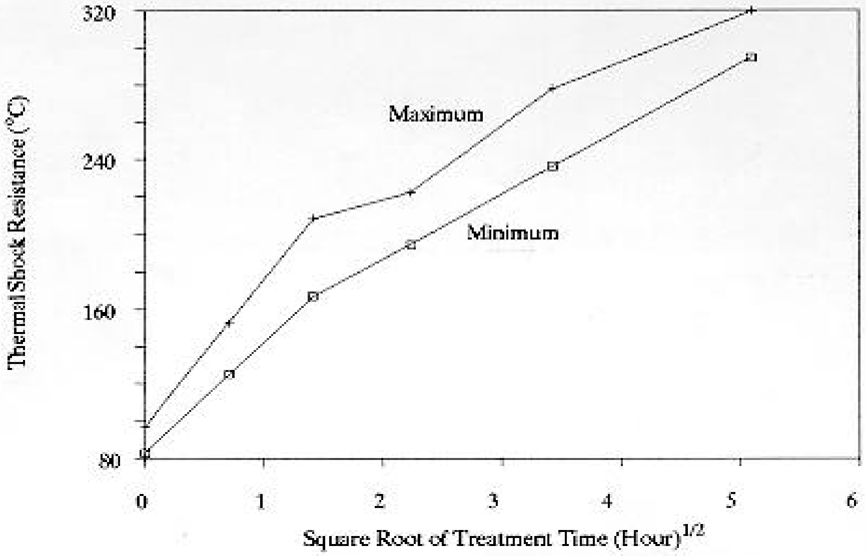

Figure 2. Thermal shock resistance of QX/Nd glass vs. square root of the treatment time in the salt bath

$$\Delta T = AP/4\pi KL \qquad (2)$$

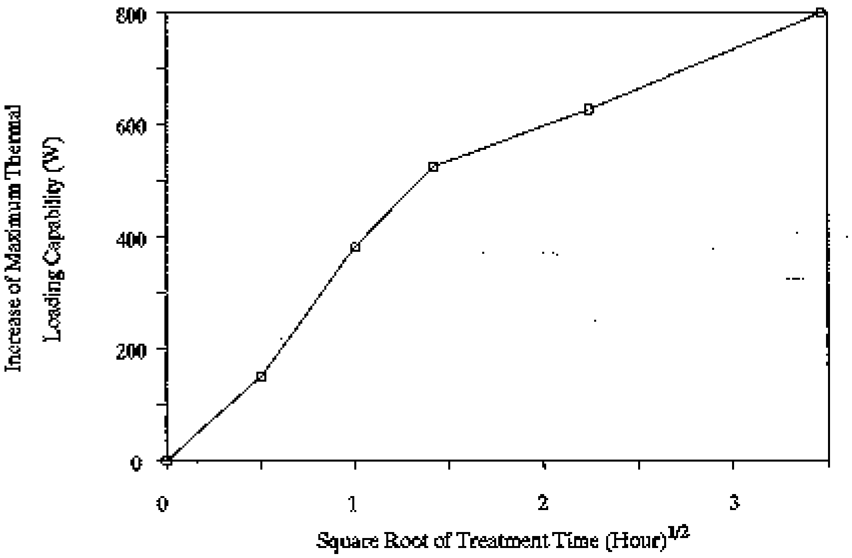

Figure 3. Improvement of maximum thermal loading capability vs. square root of treatment time in the salt bath

In equation (2), A is the fraction of the flashlamp power dissipated as heat in the glass rod, P is the flashlamp power, K is the thermal conductivity and L is the length of the rod. The parameter, A, remains a constant for a laser glass rod with selected doping concentration and configuration. The flashlamp power required to fracture the glass rod is called the maximum thermal loading. Figure 3 shows the increase of the maximum thermal loading capability of QX/Er glass for rods with a dimension of 5 mm diameter by 76 mm long treated in the salt bath for varying times. Combining equations (1) and (2), the value of A, 0.08, is derived. This is a reasonable value for QX/Er glass and in accordance with what we would expect from its absorption spectra. Since QX/Nd doped with 3 weight per cent Nd_2O_3 exhibits a lower value of A than QX/Er glass due to less absorption, the maximum thermal loading capability turns out to be much higher than that of QX/Er. Testing of the maximum thermal loading capability of QX/Nd glass was limited by the capability of the power supply used for this experiment.

The thickness of the ion-exchange layer on the glass surface is approximately 10 ~ 20µm for a 24 hourtreatment[5]. This layer should maintain its integrity if an air cooling system is utilized for the laser. However, this layer may be corroded away to some extent when it is employed in a water-cooled laser system operated over a long period of time. For a phosphate glass rod, it could be assumed that there is no buildup of insoluble reaction products on the rod surface and no change in the character of the coolant when the rod is corroded by the cooling water [7]. Thus the corrosion process proceeds at a constant rate.

Temperature, T, and corrosion rate, R, are related by the Arrhenius equation in the ideal case.

$$\mathbf{Log\ R = C/T} \qquad (3)$$

In equation (3), C is a constant reflecting the activation energy of the corrosion process. Thus, an accelerated degradation test could be carried out by immersing strengthened cylinders into deionized boiling water for various times. Samples set in the boiling water for two hours exhibit similar thermal shock resistance. By measuring the weight loss, the corrosion rate in the boiling water was found to be $5x10^{-5}$ g/cm.hour for QX/Er glass. This is equal to 160nm thickness per hour. According to equation (3), the corrosion rate at room temperature should be much smaller. This indicates that the surface compressive layer is quite stable. It should be noted that a small tension stress is built up in the interior of the rod when the compressive layer is formed on the surface. This tension stress would cause cracks in the rod when the surface compressive layer is removed.

3. LASER PERFORMANCE TEST RESULTS AND DISCUSSION

3.1 QX/Er Glass

A high average output power test was performed utilizing a chemically strengthened 3mm diameter by 80mm long rod in a Kigre RFE-663 laser head. The resonator consisted of a flat high reflector and a flat output coupler with a reflectance of 85% at 1.54µm. The discharge pulse duration was 1.0ms. Figure 4 illustrates the relationship between the laser output energy at 1.54µm and the input pump energy at a repetition rate of 10 Hz. A maximum average output power of 6.5W was achieved. Figure 4 shows that there is no obvious decrease of laser efficiency even when the lamp input power is greater than 1 kW.

Q-switched laser testing was carried out using a rotating prism from a Kigre KR-174 transmitter which is designed for flashlamp pumped eyesafe laser rangefinder. A 3mm diameter by 80 mm long QX/Er glass rod was employed. The thermal lensing in a high repetition rate Er:glass laser resonator will have a significant impact on the mode structure and polarization of the output. In order to operate in single mode, a specific resonator configuration has to be designed for each particular pump level. Various resonator configurations were designed and implemented according to the thermal lensing data generated for different pump energies and repetition rates. A single mode laser output of 5 mJ was achieved at 1.54µm with a 60ns pulse width at a repetition rate of 15 Hz.

Both the values of the maximum average output power and the repetition rate for Q-switched laser at 1.54µm represent an increase of several times comparedto previously reported Er^{3+} glass lasers . The maximum input power was 1kW for the high average power test and 300W for the high repetition rate Q-switched laser experiment. These values are still far below the maximum thermal loading capability of 1.5kW for such a chemically strengthened QX/Er glass rod. This indicates that better performance is achievable through improved laser designs.

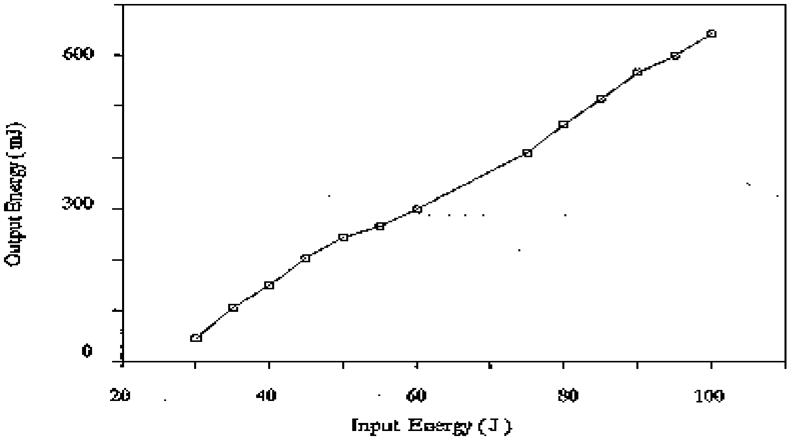

Figure 4. Laser output vs. pump energy for chemically strengthened QX/Er glass at 10 Hz repetition rate

3.2 QX/Nd Glass

A Nd^{3+} doped chemically strengthened glass laser test was conducted using a 10 mm diameter by 160 mm long rod pumped with two KS36 lamps in a Kigre FD106SK pump chamber with a discharge pulse duration was 2ms. The laser resonator consisted of a high reflector with a 10m curvature and a flat output coupler with a reflectance of 60% at 1.05μm. The pump repetition rate was fixed at 5 Hz during the initial stage of the test. The input power was increased by elevating the input voltage. After the input voltage had reached its maximum, the repetition rate was increased in order to input more power into the laser glass rod until the maximum power supply discharge capability of 3KW was reached.

Figure 5 shows that these two stages exhibit slope efficiencies of 5.4% and 3.9%, respectively. It is believed that the decrease in slope efficiency exhibited in the second test stage is a result of thermal lensing. The thermal lensing is a result of the high temperature gradient which extends from the rod center to the barrel surface for such a high power pump condition. This lensing appears although QX/Nd laser glass exhibits a low temperature coefficient of optical path length of 4.5×10^6 k^{-1}.

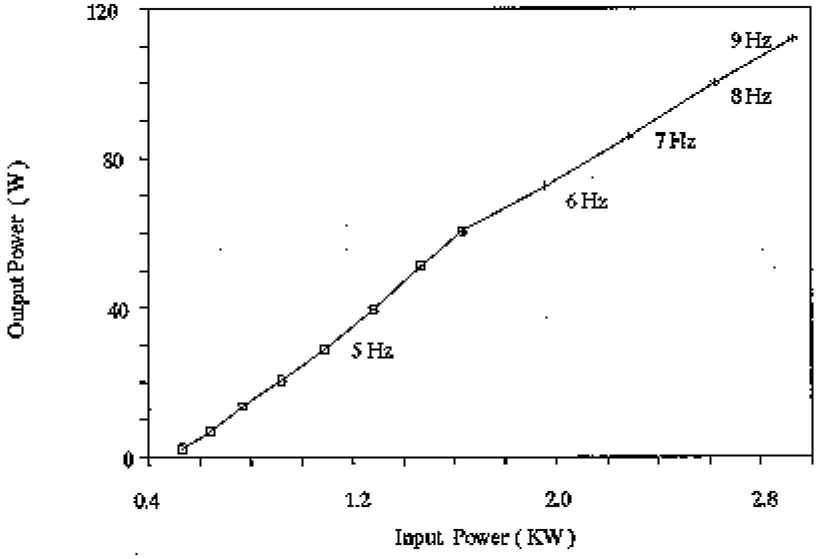

Figure 5. Laser output vs. pump power for chemically strengthened QX/Nd glass at various repetition rates

Greater than I 10W of average laser output power at 1.05µm was obtained. The maximum thermal loading capability for such a chemically strengthened rod is estimated to be greater than 5KW, indicating an average output laser power greater than 110W should be obtainable.

4. CONCLUSION

The ion-exchange chemical strengthening process for Cr^{3+}:Er^{3+} doped QX/Er and Nd^{3+} doped QX/Nd glasses has been investigated. The thermal shock resistance was improved by a factor of 2, resulting in a dramatic enhancement of the thermal loading capability for both glasses. A free running laser with an average output power of 6.5W and a Q-switched single mode laser with an energy of 5mJ and a pulse width of 60 ns at a repetition rate of 5Hz have been achieved at l.54ym utilizing a 3mm diameter by 8O m long chemically strengthened QX/Er glass rod. Greater than 110W of average power at 1.05µm has been demonstrated from a 10 mm diameter by 160 mm long QX/Nd chemically strengthened glass rod. Further improvement of laser performance for both glasses is obtainable by modifying the laser system design.

5. ACKNOWLEDGEMENT

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