DEVELOPMENT OF GLASS PHOSPHORS FOR WHITE LIGHT EMITTING DIODES

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OBJECTIVE:

The ultimate objective of this work is to create a glass phosphor that would convert blue light from a nitride-based LED into a high-quality white light spectrum that would mimic daylight and which could be used in commercial and domestic lighting. This would be done by directly applying a glass phosphor coating or cap containing rare earth phosphors to the LED to obtain a color balance. The development of the glass is based upon a combination of energy conversion and controlled scattering. Nitride-based LEDs emitting in the blue end of the spectrum were employed and borosilicate glasses doped with europium, samarium, dysprosium and terbium were applied as the phosphor coating. Different mole percentage combinations of these rare earth dopants were used to determine the composition which would best provide a white light similar in quality to daylight. Controlled scattering by possible devitrification and liquid-liquid phase separation were beneficial in achieving this. UV-visible-NIR spectroscopy, X-ray diffraction and fluorimetry were used to assess the quality of the fabricated glass. An Ocean Optics measurement system was used to examine the resulting spectrum of the capped LED. Final results concluded that the phase separated portions of the 3 mole% rare earth glass provided the best light color and quality.

INTRODUCTION:

Light Emitting Diodes have been researched as a means to reduce global energy costs due to their efficiency in converting electrical energy into light. LEDs use up to 90% less energy than incandescent bulbs because they emit light by passing electrons across a semiconductor rather than by heating a filament. LEDs generate less heat because a much higher percentage of electrical power goes directly to generating light. The potential energy savings would reduce both pollution and the dependency on fossil fuels for power generation. A study conducted by Sandia National Laboratories indicated that if 6% of conventional lighting were replaced by LED lighting, it would save over $18 billion in energy costs (1). The use of LEDs in conventional indoor lighting will provide a significant impact in energy efficiency.

PROCEDURE:

Glasses were prepared containing different mole percentages of rare earth elements in a sodium borosilicate glass. Each batch consisted of reagent grade Na_2CO_3, H_3BO_3, SiO_2 and TiO_2 with Eu_2O_3, Sm_2O_3, Dy_2O_3 and Tb_4O_7 and was melted in a platinum crucible at 1450°C. After melting for one hour, the crucible was removed from the furnace and the glass was poured into a mold formed by pre-heated steel bars, annealed at 450°C for an hour, and cooled overnight. The glasses were then polished using successively finer silicon carbide paper, to remove scratches and form smooth, parallel surfaces, and finished with a diamond polish.

Differential Scanning Calorimetry was used to determine the glass transition temperature of each of the glass samples. This information was then used in determining the temperatures to use in the vacuum furnace to try to slump the glass into a shape suitable for attaching to the LED. These slumped pieces were then placed onto the LED and the intensity and wavelengths of the emitted light were recorded using a spectroradiometric system.

Emission and excitation scans were performed using the SPEX Fluorolog 2 fluorimeter. From the varying emission intensity, it was determined which rare earth mole percentage combinations would be likely to provide the best light intensity at the desired wavelengths. The peaks from each of these scans were examined and the peak height was plotted against batched total concentration of rare earths for each wavelength.

The glass samples were placed in front of an emitting blue LED to see which colors were transmitted and whether phosphorescent conversion took place. The approximate spectrum of the blue LED was measured using an Ocean Optics spectroradiometric system to determine the peak emission. The glass samples were then run through the fluorimeter for emission scans at the excitation wavelength of 453 nm.

Heat treatment was conducted on glass samples of 1.5 mole percent total rare earth (RE) to determine the effect of possible devitrification on emission intensity. X-Ray Diffraction was then carried out on the piece heat treated at 700 degrees C to determine what crystal phase or phases had precipitated. No crystalline phases were found, so either the volume of the crystallization was too small or the glass was phase separated. Another series of heat treatment testing was carried out on a 1.5 mole percent rare earth glass. This was to determine if length of heat treatment time had an effect. The pieces were all heated at 675 degrees C for various lengths of time. With increasing dwell time, the pieces turned more opaque.

RESULTS:

Photos were taken to record the visible differences between the glass samples. The samples photographed were the initial 3 mole% RE glass from the side and from the center of the piece (where the most phase separation occurred), the 2nd 3 mole% RE batch from the center, 1.5 mole% RE, the sample of 1.5 mole% RE heat treated for $^1/_2$ hour, the 1.5 mole% sample heat treated for 1 hour, and the blue LED.

DISCUSSION:

Based on the photographed results, it was concluded that of the various tested borosilicate glasses, the glass containing 3 mole percent total rare earth was the best composition. Controlled scattering by possible devitrification and/or liquid-liquid phase separation created opacity, resulting in optimized absorption of the blue light from the LED. It is possible that allowing an increase in residence time for the blue light in the glass resulted in a greater conversion of blue light into yellow. Opacity increased as the mole percentage of rare earth elements increased, resulting in more optimal coloration and scattering of the light. However, increased opacity did present a decrease in overall intensity.

CONCLUSIONS:

The glass phosphor coating composed of the sodium borosilicate glass with 3 mole total rare earth percentage provided the most optimal results. The use of phase separation and controlled scattering was beneficial in obtaining the correct color balance. In the future, work towards modifying the amounts of each rare earth dopant in this glass composition could lead towards better intensity and color rendering.

REFERENCES:

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