

# Mechanoluminescent Materials: A New Way to Measure Stress by Light<sup>†</sup>

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**Abstract:** The monitoring of stress changes in structural components under various kinds of dynamical loading is crucial for the assessment of their integrity and lifetime. In addition to many methodologies available, such as strain gauges, optical fiber sensors, X-Ray diffraction and digital image correlation, we introduce a novel non-contact method to visualize stress distributions based on mechanoluminescence (ML). ML is a phenomenon occurring in some materials that emit light upon an applied stress level. In this paper, we develop the ML material  $(Ca_{0.4}Sr_{0.6})Al_2Si_2Os:Eu^{2+},Ho^{3+}$ , a glow-in-the-dark material, to visualize stress distribution in a disc, as well as the stress field of an ultrasonic transducer. The properties of defects in the ML phosphors, which are responsible for ML in this material, are vital for stress visualization.

**Keywords:** mechanoluminescence; anorthite; stress visualization; acoustically induced piezoluminescence; Von Mises stress.

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## 1. Introduction

Structural components undergo a variety of dynamic loading conditions during their operating life. The monitoring of the mechanical stresses induced by such loading is vital for the assessment of the components' structural integrity [1]. Large and abrupt stress changes and stress concentrations can in fact be responsible for the onset of micro-cracks in the component and may drastically decrease the component residual strength as well as its life expectancy. Many techniques for stress monitoring are available, using both contact and non-contact sensing devices and methodologies. The use of strain gauges is very common and accessible for many applications, requiring simple wiring. Another contact method is to use Fiber Bragg Grating (FBG) sensors, which shift the wavelength of reflected light as the fibers are strained [2]. Non-contact techniques for measuring stress distribution are also available. Digital image correlation is a popular one, but it requires relatively expensive setups [3]. Photoelasticity also plays a role in visualizing stress distribution, because the difference of maximum principal stress and minimum principal stress is proportional to the isochromatic value [4]. It is applicable to complex geometry, but is limited to transparent objects and polarized light is required.

Mechanoluminescence is an alternative route to visualize stress distribution. ML materials are special phosphors which can emit light when subjected to mechanical loads. The intensity of the emitted light is directly linked to the intensity of the stress field, which can therefore be determined by measuring and adequately processing the ML phenomenon [5]. Interestingly, even the stress field introduced by other excitation sources, such as inverse piezoelectricity, magnetorestriction and

ultrasound irradiation can be visualized by ML phosphors. This has expanded the application range of ML [6]. Although many different phosphors can readily be prepared by doping transparent crystals with luminescent ions, the number of nondestructive ML phosphors is still below 100, which limits the tuning of desired properties such as emission color, sensitivity to load, afterglow, etc. [6]. To optimize the ML effect for certain applications, engineering and tailoring of ML phosphors are crucial. In this paper we report about the anorthite  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:\text{Eu}^{2+},\text{Ho}^{3+}$  ML material, which is responsive to both mechanical loading and ultrasonic stimulation. We show that the inclusion of  $\text{Ho}^{3+}$  improves the ML intensity and that the concentration of defects and their energy position with respect to that of the pure host crystals are very important for tuning the ML properties.

## 2. Materials and Methods

ML materials  $\text{ZnS}:1\%\text{Mn}$ ,  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$  and  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  were prepared by a solid-state reaction method, i.e. sintering the starting materials under a high temperature. Starting materials were weighted and ground in an agate mortar for 10 minutes and then were pressed into discs of 16 mm diameter. Those discs were placed in a corundum crucible, which was then placed in a horizontal tube furnace and heated at 1050 °C for 3 hours under a gas flow of  $\text{N}_2$  (flow rate 60 ml/min) for  $\text{ZnS}:1\%\text{Mn}^{2+}$ , and at 1350 °C for 6 hours under 95% $\text{N}_2$ -5% $\text{H}_2$  (flow rate 140 ml/min) for  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:\text{Eu}^{2+},\text{Ho}^{3+}$ . After cooling down to room temperature, the discs were crushed and ground into fine powders for further characterization.

The emission and excitation spectra were collected with an Edinburg Instrument FS920 spectrometer at room temperature. For thermoluminescence measurements, the phosphor powders were pressed into small discs of roughly 5 mm diameter. The disc was placed on a heating stage in a vacuum chamber. After exciting the disc with a UV light with a wavelength of 370 nm from a LED (for  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$ ) and with 300 nm UV light from a Xenon lamp (for  $\text{ZnS}:1\%\text{Mn}$ ) for 10 minutes, the discs were cooled down to -60 °C and then heated up to 225 °C with a fixed rate of 0.5 K/sec. The luminescence was detected with a calibrated photometer ILT1700, and corrected for thermal quenching (i.e. the drop of photoluminescence occurring as the temperature increases).

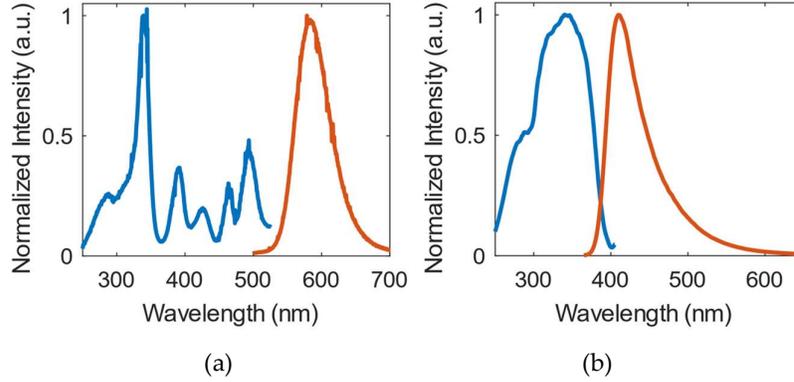
For the stress visualization, coupons (length > 120 mm, width 12.5 mm, thickness 2.0 mm) were prepared by the resin transfer molding method. Resin (Epikote™ Resin MGS LR 135) and hardener (Epikote™ Resin MGS LR 137) were taken in a 10:3 weight ratio and mixed by stirring together with ML powders (weight ratio 3%). Then they were infused through a mold, cured at room temperature for 24 hours, post-cured at 85 °C for 15 hours and then cooled down to room temperature for demolding. These coupons were then placed inside an Instron ElectroPuls E10000 apparatus for tensile tests. After illuminating with UV light (356 nm) for 150 seconds, each coupon was left unloaded for a period to allow the relaxation of the strong afterglow. Then a linear tensile load was applied, and the maximum load was held for at least 200 seconds before being released. The light emission was collected with a photomultiplier tube (Hamamatsu® H10722-210) and synchronized with displacement and force signals via a National Instrument® data acquisition unit (NI 9234) with a sampling rate of 2048 Hz.

Besides these coupons, a disc (diameter 25 mm, thickness 10 mm) of epoxy resin (Struers® Epofix®) mixed with ~0.3 g  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  was compressed using a hydraulic machine (AC Hydraulic™) while the ML was recorded with an CMOS camera (Ximea® MC031MG). The ultrasonic field was also visualized by a thin epoxy plate embedded with ~0.1 g  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  (diameter 30 mm, thickness ~0.5 mm), which was irradiated with ultrasound from a transducer (Gymna Pulson 200) placed at a distance of 70 mm from the sample while the emission pattern was recorded with the CMOS camera.

## 3. Results

The solid-state synthesis yielded pure phases of the investigated ML materials (X-Ray diffraction patterns not shown here). As can be seen from figure 1a,  $\text{ZnS}:1\%\text{Mn}^{2+}$  can be excited efficiently by wavelengths up to 500 nm, and its emission band is centered at 583 nm, which results in a yellow-

orange color.  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  can be excited by UV light from 250-400 nm and the emission band is centered at 411 nm, which corresponds to a blue color (Figure 1b). Because of their non-centrosymmetric crystal structures both phosphors show piezoelectricity, i.e. an internal electric field arises due to the application of stress.  $\text{ZnS}:1\%\text{Mn}^{2+}$  is very sensitive to friction and is applicable in dynamic pressure mapping, especially above 10 MPa [7], but the response to extension was not reported yet, which could reveal interesting physics of ML.



**Figure 1.** The photoluminescence emission (orange) and excitation spectra (blue) of (a)  $\text{ZnS}:1\%\text{Mn}^{2+}$  and (b)  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$ . Both phosphors are excitable at 365 nm.

### 3.1. Mechanoluminescence as a visualization of stress fields

The tests on phosphor-embedded polymer discs (radius  $R$ , thickness  $l$ ) display a clear ML pattern, as shown in in Figure 2a, where the asymmetry of the pattern is due to the imperfect loading conditions. The theoretical stress field under a diametric loading pair  $P$  (along  $y$ ,  $-y$  orientations), is as follows [8]:

$$\sigma_x = -2P/(\pi l)\{(R-y)x^2/[(R-y)^2+x^2]^2 + (R+y)x^2/[(R+y)^2+x^2]^2 - 0.5/R\}, \quad (1)$$

$$\sigma_y = -2P/(\pi l)\{(R-y)^3/[(R-y)^2+x^2]^2 + (R+y)^3/[(R+y)^2+x^2]^2 - 0.5/R\}, \quad (2)$$

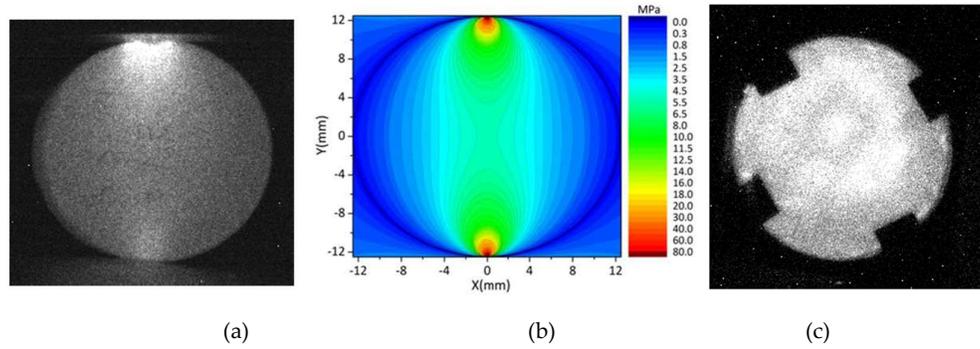
$$\tau_{xy} = 2P/(\pi l)\{(R-y)x^2/[(R-y)^2+x^2]^2 - (R+y)x^2/[(R+y)^2+x^2]^2\} \quad (3)$$

and the Von Mises stress  $\sigma_{VM}$  is defined as:

$$\sigma_{VM} = [\sigma_x^2 + \sigma_y^2 - \sigma_x\sigma_y + 3\tau_{xy}^2]^{0.5} \quad (4)$$

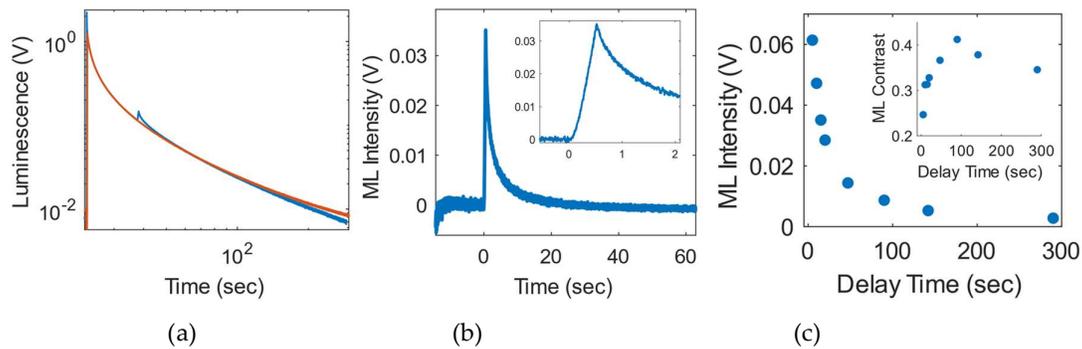
From Figure 2b, the ML pattern agrees with the distribution of  $\sigma_{VM}$ , which indicates that the deformation energy plays a vital role in inducing ML. Actually,  $\sigma_{VM}$  is directly related to the stress deviator,  $S_{ij} = \sigma_{ij} - \Sigma\sigma_{ii}/3$  through  $\sigma_{VM} = (1.5S_{ij}\cdot S_{ij})^{0.5}$ , which is responsible for the change of shape of materials [9]. On the other hand, the hydraulic stress  $\Sigma\sigma_{ii}/3$  is responsible for the change of volume. Thus, it is possible that mechanical manipulation of ML phosphors excites various defects, such as point defects, dislocations, faults, etc., due to the application of stress, and then releases electrons that were stored during the excitation by UV light.

It is interesting to note that  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  is also able to visualize the stress field produced by ultrasonic stimulation, i.e. Acoustically induced PiezoLuminescence (APL), as shown in Figure 2c. The ring and circle at the center of the image correspond to ultrasound-induced stress at the positions of irradiation. The asymmetry is due to the imperfection of the transducer. This effect lasted tens of seconds during the ultrasonic radiation, but disappeared in 1-2 seconds after the ultrasound was removed.



**Figure 2.**  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  displays (a) ML under compression and (b) its pattern agrees with the Von Mises stress from calculation, and (c) APL, where the ring+circle pattern is the emission of light due to ultrasonic stimulation.

The visualization of stress fields heavily depends on the linearity between stress and ML. This linearity can be readily confirmed in a series of tensile experiments. It is worth noting that the afterglow should be subtracted from the observed emission profile, which is the compound of net ML and afterglow, as shown in Figure 3a. A net ML profile can be obtained, in Figure 3b, and a linear relationship of ML with time (and thus with force) is observed between  $t=0$  and  $t=0.5$  seconds, during which extension increased from 0.00 to 0.60 mm at a rate of 1.2 mm/sec. A long tail up to tens of seconds prevails after the peak of ML, which is related to the combination of recombination with luminescent centers and re-trapping at defects of electrons liberated due to stress. The delay time between the end of the UV excitation and the application of the load influences the ML magnitude greatly. In Figure 3c, the magnitude of the ML peak drops very quickly as the delay time is increased. However, the afterglow also decays with time, resulting in the maximum ML contrast, i.e. ML/afterglow (inset of Figure 3c), after a delay of 90 secs. Therefore, for visualizing stress distribution, it is important to incorporate a delay time in order to reach a good compromise between ML intensity and ML contrast.



**Figure 3.** For tensile tests of  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$ , (a) the magnitude of ML+afterglow (blue) drops below its afterglow (orange) at a certain time, and (b) the net ML (delay 15 sec) lasts tens of seconds and is linear with time in the [0 0.50 sec] interval (inset) where the load is applied linearly, (c) The peak of net ML decreases with delay time, leading to a maximal ML contrast at a delay of 90 seconds (the inset).

### 3.2. Trap Properties as a Mean to Model Mecholuminescence

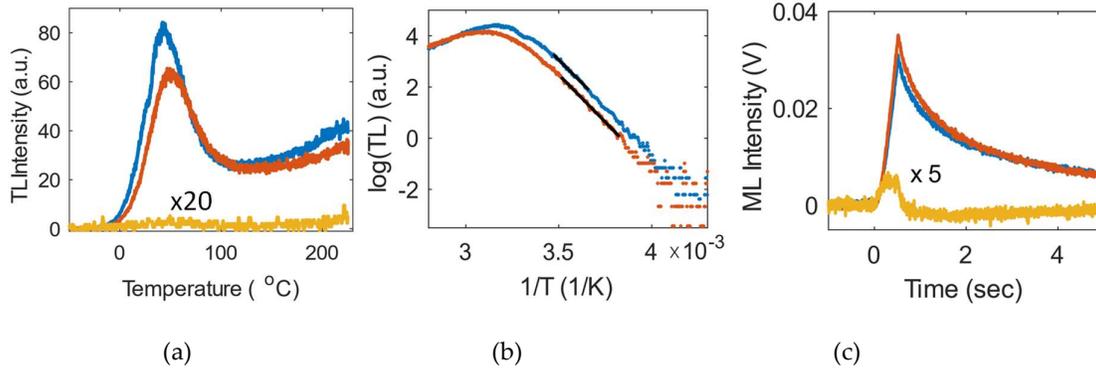
The regular packing of atoms in crystals makes it possible to describe the energy of electrons with a certain wave vector  $\mathbf{k}$ , i.e.  $E(\mathbf{k})$  in a band diagram. Here, the electrons of filled orbitals form the valence band, which is separated by the bandgap from the conduction band, where electrons can move freely in the entire crystal if they are excited above the band gap. However, doping with impurity ions such as  $\text{Mn}^{2+}$  or  $\text{Eu}^{2+}$  leads to additional energy levels inside the band gap. Special synthesis procedures (such as sintering in reducing gas,  $\text{H}_2$ ) will create additional defects, which are

able to donate/accept charge carriers to/from the crystal and even trap these charge carriers, for example in glow-in-the-dark phosphors. The concentration of traps  $n$  and the energy levels with respect to the conduction band minimum, i.e. the trap depth  $E_{\text{trap}}$ , play an important role. For example, electrons trapped at defects can escape at a rate  $p=s \cdot \exp(-E_{\text{trap}}/kT)$ , contributing  $n \cdot p$  to the change of afterglow in many cases, in which  $k$  is the Boltzmann constant,  $s$  an attempt-to-escape frequency and  $T$  the temperature. Thermoluminescence (TL), i.e. the study of luminescent intensity as a function of temperature while heating at a fixed rate ( $dT/dt = \beta$ ), is a very useful tool to reveal the trap depths and trap concentration. In a TL profile, a distinct glow peak profile (at a certain temperature  $T_m$ ) can correspond to a trap depth  $E_{\text{trap}}$ , and the area under the curve is proportional to the trap concentration. Various methods are available to calculate the trap depths, see [10] for a review.

Among these methods, the initial rise method is generally applicable to extract trap depth and does not depend on the kinetics of the detrapping mechanism of electrons [11]. It is assumed that, at the early rise of a TL peak, the rate of electrons escaping from a trap is negligible and the intensity of TL follows from

$$I_{\text{TL}} = C \cdot \exp[-E_{\text{trap}}/(kT)], \quad (5)$$

where  $k$  is the Boltzmann constant,  $T$  the temperature and  $C$  a constant.



**Figure 4.** (a) The TL spectra of  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$  (blue),  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  (orange) and  $\text{ZnS}:1\% \text{Mn}^{2+}$  (yellow,  $\times 20$ ) and (b) their corresponding fitting (black) by initial rise method, and (c) the ML profiles for a delay time of 15 secs, except for  $\text{ZnS}:1\% \text{Mn}^{2+}$  (5 seconds,  $\times 5$ ).

For  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$ , the introduction of 1% of  $\text{Ho}^{3+}$  lowered the height of the TL peak in Figure 4a, which indicates a lower trap concentration. It also shifted the peak temperature  $T_m$  to higher values, and indeed the extracted trap depth increased from 0.623 eV (for  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$ ) to 0.638 eV as calculated from the fitting of the linear part (below 20% of the intensity at  $T_m$ ) of the  $\log(I_{\text{TL}})-1/T$  plot in Figure 4b. Interestingly, the TL of  $\text{ZnS}:1\%\text{Mn}^{2+}$  was negligible, under excitation by 302 nm (excitation of the ZnS host) or 365 nm (exciting mainly  $\text{Mn}^{2+}$ ), which meant that the trap concentration was very low, or the trap depth was so small that all traps were already emptied before the TL started [12]. It is notable that the ML of  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$  is higher than that of  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$ , and  $\text{ZnS}:1\%\text{Mn}^{2+}$  only show limited ML under tensile extension although very efficient ML due to friction, scratching or compression of single crystals have been reported [13]. Normally, ML is due to the release of trapped electrons. However, these trapped electrons may have a different trap depth distribution from the traps responsible for afterglow. For example, deeper traps can yield ML where afterglow is minimal (a delay of 300 sec still can provide ML in Figure 3c). Judging from Figure 4a-c, it is concluded that the traps in  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$  responsible for ML are situated in the deeper part of the trap depth distribution. Therefore, the introduction of  $\text{Ho}^{3+}$  enhances ML as it shifts the traps to larger depth although the trap concentration is only slightly decreased. The contrast of images that visualize stress are hereby improved since the ratio of ML/Afterglow is increased here from 0.15 (for  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}$ ) to 0.28 (for  $(\text{Ca}_{0.4}\text{Sr}_{0.6})\text{Al}_2\text{Si}_2\text{O}_8:1\%\text{Eu}^{2+}, 1\%\text{Ho}^{3+}$ ). The combination of a reduction of trap concentration and an increase of trap depth is responsible for the improvement.

This indicates that tuning the trap depths and trap concentration is a useful way to tune the properties of ML according to specific applications such as stress visualization, APL, or ML displays. This is possible when the traps responsible for ML are already known for the studied materials.

#### 4. Conclusion

Mechanoluminescent phosphors offer an alternative technique for stress visualization based on the linearity between stress and mechanoluminescence. The anorthite ( $\text{Ca}_{0.4}\text{Sr}_{0.6}\text{Al}_2\text{Si}_2\text{O}_8:\text{Eu}^{2+},\text{Ho}^{3+}$ ) is applicable for stress visualization and APL visualization. The inclusion of  $\text{Ho}^{3+}$  shifts the trap depth to larger values and slightly lowers the trap concentration, thus increasing the ML intensity and the contrast of ML to afterglow. Tailoring trap properties is vital for tuning ML phosphors for various applications.

**Author Contributions:** Ang Feng and Philippe F. Smet designed the experiments. Ang Feng prepared the ML phosphors, collected photoluminescence spectra, and TL profiles. Alfredo Lamberti and Ang Feng prepared the coupons, and collected ML profile. Simon Michels assisted with the APL experiments. Ang Feng analyzed the data and wrote the first draft of the paper, and all authors revised the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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