

KMgF₃:Eu²⁺ as a new fluorescence-based pressure sensor for diamond anvil cell experiments

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ABSTRACT

We present an optical pressure sensor KMgF₃:Eu²⁺ suitable for experiments in diamond anvil cell (DAC), which we have calibrated against the ruby reference sensor at pressures ranging from atmospheric up to 30 GPa (300 kbar), at room temperature. The emission spectra of KMgF₃:Eu²⁺ consist of one prominent emission line related to the ⁶P_{7/2} → ⁸S_{7/2} electronic transition (zero phonon line) accompanied by weak phonon sidebands. The energy of emission at atmospheric pressure was determined to be 27846 ± 7 cm⁻¹. The edge of the excitation spectrum lies at wavelength around 325 nm (30800 cm⁻¹) making it suitable to excite with He-Cd laser (325 nm). The sensor exhibits linear pressure shift of rate equal to -0.815 ± 0.007 cm⁻¹/kbar at pressures up to 300 kbar. The pressure shift of the emission is totally reversible and the pressure sensor is suitable for multi cycle pressure experiments. The sensor's exceptional feature is that excitation and emission spectra lie in near UV, which makes it very convenient to use in high pressure optical measurements in visible and IR region with no interference from the sensor's luminescence.

1. Introduction

Determination of the value of high hydrostatic pressure in the diamond anvil cell (DAC) is performed by the measurements of pressure induced changes of magnitudes of physical parameters of a known system (pressure sensor) encapsulated in the DAC together with the investigated sample. Established methods include measurement of lattice constant of known equation of state (e.g. Au, NaCl) by means of X-Ray diffractometry [1] as well as Raman shift of compressed pressure sensing material or the diamond anvil itself [2]. However, the most popular is the spectroscopic method based on pressure induced changes (shifts) of absorption or/and emission spectra of certain compounds. Usually pressure induced shift of the fluorescence lines of the specific material is related to the value of pressure.

Materials selected for luminescence pressure sensor should be characterized by strong intensity of the emission, stable at broad range of temperatures and which transition energy changes linearly with pressure. It is also important that the emission of the sensor does not overlap with the emission of the sample. The last requirement imposes continuous search for new pressure sensors suitable for specific spectral region.

By far the most commonly used standard is the luminescence of the

R₁ and R₂ spectral lines of ruby (Al₂O₃:Cr³⁺) thanks to large pressure shift and emission brightness [3–5]. The method was first proposed by Forman *et. al* [3], and later continuous effort to calibrate of the ruby sensor for ever higher pressures was carried out resulting in extension of the ruby fluorescence method to sensing pressures reaching 1.5 Mbar [6–8]. However, ruby pressure sensor suffers from some considerable disadvantages. These include temperature dependence of emission wavelength, line broadening at high temperature and high or non-hydrostatic pressure causing overlap of the ruby doublet as well as quenching of the ruby luminescence at high temperature or pressure [5,9]. These issues prevent using ruby in some applications and triggered search for alternative pressure sensing materials.

Alternative materials for spectral pressure sensors, proposed to eliminate some of the aforementioned issues include materials activated with: Cr³⁺ (most notably alexandrite (BeAl₂O₄:Cr³⁺) [10]), Sm²⁺ (most notably SrB₄O₇:Sm²⁺ [9,11] and YAG:Sm²⁺ [12–15]) as well as other rare-earth ions [16–18]. Extensive discussion on various optical pressure sensors can be found in Ref. [19]. Recently a new type of pressure gauges, based on luminescence of InAsP semiconductor quantum wells, has been proposed [20]. Common feature to the above mentioned materials is that their luminescence spectra lie in red and near infrared region - 693 nm for ruby, 640–760 nm for alexandrite

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[21], 680–740 nm for SrB₄O₇:Sm²⁺ [11] and 550–730 for YAG:Sm²⁺ [12] at ambient pressure. This poses a difficulty for optical measurements (luminescent spectra, lifetimes etc.) in that spectral region due to possible overlap of emission spectra of measured sample and pressure sensor, which not always are easy to separate. In case of weakly luminescent samples this may render the sensor inapplicable.

To address this issue we propose KMgF₃:Eu²⁺ as luminescent pressure sensor. KMgF₃:Eu²⁺ is one of the few Eu²⁺ activated materials where electronic levels of the 4f⁶5d configuration lie significantly higher than the excited states of 4f⁷ configuration. Such materials instead of the typical broadband d-f emission show f-f emission in the form of narrow lines. The KMgF₃:Eu²⁺ luminescence consists of one strong line attributed to ⁶P_{7/2} → ⁸S_{7/2} transition (zero phonon line), which at ambient pressure is situated around 359 nm [22] accompanied by weaker phonon sidebands. The KMgF₃:Eu²⁺ emission line exhibits linear shift with pressure up to at least 300 kbar without any significant quenching or mixing with the 4f⁶5d¹ configuration levels as evidenced by stable decay lifetimes with respect to the pressure [23]. KMgF₃ host crystallises in cubic perovskite structure, it shows no structural changes at temperatures 4–1073 K [24] and at pressures up to 500 kbar [25]. For this reason undoped KMgF₃ has also been suggested as a X-ray pressure calibrant [24]. The wavelength of the KMgF₃:Eu²⁺ emission in the UV region, without any emission in the visible and IR region, makes the KMgF₃:Eu²⁺ a very perspective materials for pressure sensor in spectroscopic experiments in the DAC.

2. Experimental

KMgF₃ single crystals doped with Eu²⁺ ions concentration were grown from the melt in Ar gas atmosphere by Czochralski method. Details of growing procedure are described in the paper [26].

The high pressure luminescence measurements were performed in a Merrill-Bassett type screw-driven diamond anvil cell (DAC) [27]. The DAC featured two type IIa synthetic diamonds with sixteen-sided cut. The diameter of the culet (working surface) was 0.5 mm. The gasket for the pressure chamber was made from 0.25 mm Inconel X-750 sheet preindented to 0.075 mm. A hole with diameter 0.2 mm was drilled in the centre of the indentation. KMgF₃:Eu²⁺ sample and reference ruby pressure sensor were loaded inside the hole and next immersed in polydimethylsiloxane oil acting as a pressure transmitting medium.

High pressure luminescence emission spectra of KMgF₃:Eu²⁺ were acquired using a 0.75 m focal length Czerny-Turner grating spectrometer Shamrock SR750 D1 with an iDus 420 CCD detector (Andor Technology). The measurements were performed using a 1200 lines/mm reflection grating. The sensor's luminescence was excited using K5352R-D He-Cd CW laser (Kimmon Koha) operating at 325 nm. The high pressure luminescence spectra of ruby reference sensor were acquired using a 2 m focal length PGS-2 Czerny-Turner grating spectrometer (Carl-Zeiss Jena) equipped with R928P photomultiplier (Hamamatsu Photonics). The measurements were performed using a 650 lines/mm diffraction grating. The excitation light source for ruby reference sensor was a diode laser operating at 473 nm.

High pressure luminescence excitation spectra were performed using self-made spectrofluorometer. The excitation part of the apparatus consists of 150 W xenon lamp, SPM2 monochromator (Carl Zeiss Jena) and R928P photomultiplier for lamp reference measurement. The detection part consists of SPM2 monochromator and R928P photomultiplier as signal detector.

3. Results and discussion

Fig. 1 presents pressure dependent excitation spectra of KMgF₃:Eu²⁺ observed at emission wavelength of the ⁶P_{7/2} → ⁸S_{7/2} electronic transition. The observed, unresolved structure is attributed to crystal field splitted levels of 4f⁶5d electronic configuration. The edge of luminescence excitation spectra, corresponding to the lowest

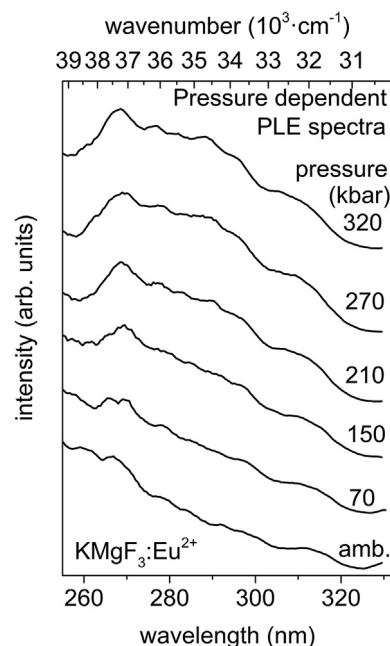


Fig. 1. Pressure dependent luminescence excitation spectra of KMgF₃:Eu²⁺ observed at ⁶P_{7/2} → ⁸S_{7/2} main emission wavelength.

electronic level of the 4f⁶5d configuration, lies around 330 nm. One should notice the peculiar pressure behaviour of the excitation spectrum. The edge of the lowest excitation band shifts very slightly towards higher energies which is contrary to the usual red shift resulting from decrease of energy of the lowest 4f⁶5d level due to increasing crystal field splitting of 4f⁶5d configuration levels. However, up to 300 kbar the material is suitable to use with He-Cd laser as excitation light source with wavelength of 325 nm.

Fig. 2 presents room temperature luminescence emission spectra of KMgF₃ doped with 0.001, 0.5 and 2.5 at. % of Eu²⁺, excited by 325 nm laser. The location of main emission peak is (for all practical purposes) the same regardless of the concentration of the compound as evidenced by the inset in the figure, which presents the location of main emission peak of respective emission spectra obtained from Gaussian fitting. The

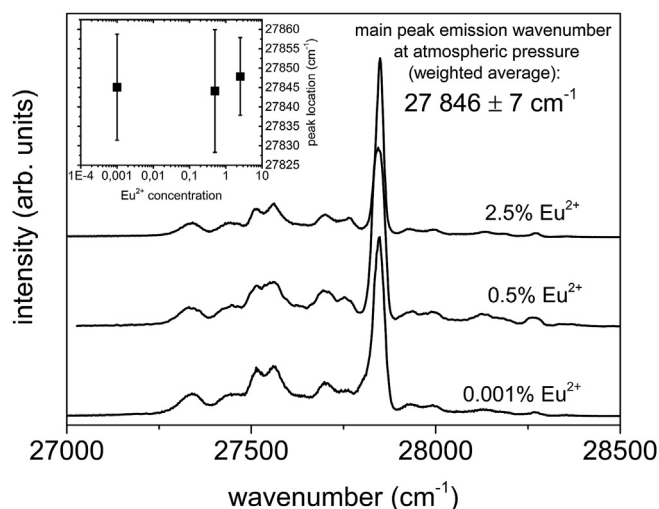


Fig. 2. Room temperature luminescence emission spectra of KMgF₃ with various concentration of Eu²⁺ dopant. The inset presents location of main emission peak of respective emission spectra. The absolute uncertainty was assumed to be equal to the HWHM values of respective peaks. The final value of emission wavenumber was calculated as the weighted average of values for each concentration.

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