

# LUMINESCENCE PROPERTIES OF $\text{Eu}^{2+}$ DOPED DIBARIUM MAGNESIUM DISILICATE, $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$

TUOMAS AITASALO, JORMA HÖLSÄ, TANELI LAAMANEN, MIKA LASTUSAARI,  
LAURA LEHTO, JANNE NIITTYKOSKI, FABIENNE PELLÉ\*

*University of Turku, Department of Chemistry, Laboratory of Inorganic Chemistry, FI-20014 Turku, Finland  
Graduate School of Materials Research, Turku, Finland*

*\*CNRS, UMR 7574, ENSCP, Laboratoire de Chimie Appliquée de l'Etat Solide,  
11, r. P. et M. Curie, F-75231 Paris cedex 05, France*

E-mail: japeni@utu.fi

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*A new persistent luminescence material, monoclinic  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$ , was prepared by a solid state reaction. Both the UV excited and persistent luminescence are observed at the green region centred at 505 nm. Both luminescence phenomena are due to the same  $\text{Eu}^{2+}$  ion occupying the single  $\text{Ba}^{2+}$  site in the lattice. The  $\text{R}^{3+}$  co-doping (R is rare earth) usually enhances the persistent luminescence of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$ . Especially, the  $\text{Tm}^{3+}$  ion has a very strong effect. Two thermoluminescence peaks at 90 and 130°C are observed in the glow curve of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$ , which is nearly ideal for a persistent luminescence material. The persistent luminescence is probably due to lattice defects i.e. oxygen (cation) vacancies, which create trapping levels for electrons (holes). Recombination of the electron-hole pair and energy transfer to the  $\text{Eu}^{2+}$  ion complete the probable persistent luminescence mechanism. The details of the mechanism(s) need, however, more experimental work to be carried out. The stable disilicate materials are very good candidates for the persistent luminescence materials as alternatives to the  $\text{Eu}^{2+}$  doped alkaline earth aluminates.*

## INTRODUCTION

Silicates are efficient luminescent materials, mainly because of their rigid and very stable crystal structures. Several silicate materials doped with rare earth ( $\text{R}^{n+}$ ) ions, either divalent or trivalent, have been proposed for or used as commercial phosphors in tricolor fluorescent lamps, scintillators *etc.* [1]. Despite their excellent host matrix properties, the silicates are not completely free of lattice defects which may decrease drastically the luminescent efficiency or cause undesired properties *e.g.* afterglow as is the case with the  $\text{Ce}^{3+}$  doped rare earth oxyorthosilicates ( $\text{R}_2\text{SiO}_5:\text{Ce}^{3+}$ ; R = Y, Lu) [2, 3].

On the other hand, the afterglow (*i.e.* persistent luminescence) can be used for such applications as security devices and exit signalization. In the mid 1990's, alkaline earth aluminates doped with the  $\text{Eu}^{2+}$  and  $\text{R}^{3+}$  ions ( $\text{MA}_2\text{O}_4:\text{Eu}^{2+},\text{R}^{3+}$ ; M = Ca, Sr; R = *e.g.* Nd, Dy) were introduced as new commercial persistent luminescence materials [4]. The luminescence properties of the aluminates may be degraded when exposed to water and hence their use in *e.g.* luminous paints as a pigment is limited. Recently,  $\text{Eu}^{2+}$  doped alkaline earth magnesium disilicates ( $\text{M}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{R}^{3+}$ ; M = Ca, Sr, Ba; R = Nd, Dy) have been found to show persistent luminescence [5-9]. A systematic study of the persistent luminescence properties, especially the effect of the  $\text{R}^{3+}$

co-doping, of all the disilicates is, however, lacking. In previous reports, the  $\text{Eu}^{2+}$  doped dibarium magnesium disilicates have been noted only very briefly [6], even if the luminescence spectrum ( $\lambda_{\text{max}} = 505 \text{ nm}$ ) is ideal for the human eye making the materials very feasible to practical applications.

In the present work, dibarium magnesium disilicates ( $\text{Ba}_2\text{MgSi}_2\text{O}_7$ ) doped with  $\text{Eu}^{2+}$  and co-doped with  $\text{R}^{3+}$  ions (R = La-Nd, Sm, Gd-Lu, Y) were prepared by a solid state reaction. The preparation and crystal structure were studied by thermal analysis and X-ray powder diffraction, respectively. The UV excited and persistent luminescence as well as thermoluminescence properties of the  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{R}^{3+}$  materials including the effect of the co-doping with the whole series of the  $\text{R}^{3+}$  ions were investigated, too.

## EXPERIMENTAL

### Sample preparation

Polycrystalline  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},(\text{R}^{3+})$  were prepared by a solid state reaction between barium carbonate ( $\text{BaCO}_3$ ), magnesium nitrate ( $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ),

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fumed silica ( $\text{SiO}_2$ ) and rare earth oxides (usually  $\text{R}_2\text{O}_3$ ). The nominal concentration of the  $\text{Eu}^{2+}$  and  $\text{R}^{3+}$  ions was one mole per cent. The starting materials were ground using a Phillips PW 4018/00 MiniMill for 15 min with a rotating speed of 3400 rpm to form a homogenous mixture. Silicon nitride grinding bowl (45  $\text{cm}^3$ ) and balls (five) were used. Sample contamination due to grinding was not observed. The powder mixture was pressed to a pellet and annealed in a reducing ( $\text{N}_2 + 12$  per cent  $\text{H}_2$ ) atmosphere for one and five hours at 700 and 1250°C, respectively.

### Thermal analysis

The investigation of the solid state reaction by thermal analysis was carried out with a TA Instruments SDT 2960 Simultaneous DTA-TGA apparatus between 25 and 1450°C. The heating rate was 5°C/min and the gas ( $\text{N}_2 + 12$  per cent  $\text{H}_2$ ) flow 100  $\text{cm}^3/\text{min}$ . The sample weight was ca. 10 mg.

### X-ray powder diffraction

The X-ray powder diffraction (XRD) patterns were collected at room temperature between 4 and 100 degrees (in  $2\theta$ ) with a Huber 670 image plate Guinier-camera ( $\text{CuK}_{\alpha 1}$  radiation, 1.5406 Å). The data collection time was 15 min. The image plate was scanned 10 times.

### Luminescence

The UV excited and persistent luminescence spectra were measured at 295 K with a Perkin Elmer LS-5 apparatus. The excitation source was a low power xenon lamp ( $\lambda_{\text{exc}} = 350$  nm). Prior to the persistent luminescence measurements, the materials were exposed to radiation from a 9W UVP Mineralight SL2537 short-wave UV lamp ( $\lambda_{\text{exc}} = 250\text{--}440$  nm).

## RESULTS AND DISCUSSION

### Preparation

A five per cent weight loss is observed in the TG curve below 200°C due to the dehydration of the magnesium nitrate (figure 1). A fraction (four moles) of water in  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  obviously evaporated already during the ball-milling, since the five per cent weight loss corresponds only to a loss of two moles of  $\text{H}_2\text{O}$ . A further multi-step 28 per cent weight loss observed

between 300 and 600°C is due to the decomposition of the magnesium nitrate and barium carbonate. The total 33 per cent weight loss corresponds very well to the calculated one (33 per cent), if four moles of water is supposed to be evaporated during the ball-milling.

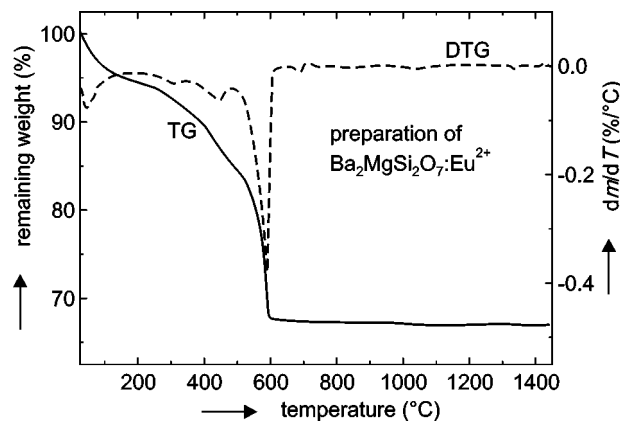


Figure 1. Thermogravimetric (TG, DTG) analysis of the solid state preparation of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$  (heating rate: 5°C/min; gas ( $\text{N}_2 + 12$  per cent  $\text{H}_2$ ) flow rate: 100  $\text{cm}^3/\text{min}$ ; sample weight: 10 mg).

### Structure

All the  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$ , ( $\text{R}^{3+}$ ) samples crystallized in the monoclinic form of  $\text{Ba}_2\text{MgSi}_2\text{O}_7$  (figure 2). This structure, with the space group of  $\text{C}2/c$  (No. 15),  $Z = 4$ , has been solved very recently [10]. The layered structure, with the following cell parameters:  $a = 8.413$  Å,  $b = 10.710$  Å,  $c = 8.439$  Å and  $\beta = 110.7^\circ$ , consists of discrete  $[\text{Si}_2\text{O}_7]^{6-}$  units connected by tetrahedrally coordinated

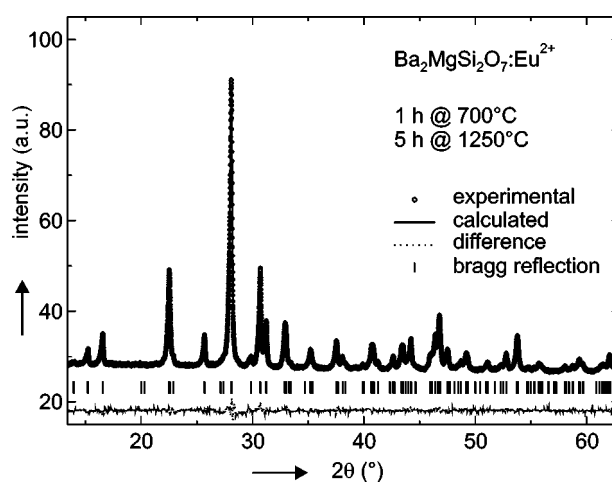


Figure 2. Experimental and calculated [10] X-ray powder diffraction patterns of the monoclinic  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$  at 295 K.

minated Mg<sup>2+</sup> and eight-coordinated Ba<sup>2+</sup> ions. No trace of the previously reported tetragonal Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub> phase [11] could be observed in the XRD patterns. In some cases, small amounts of Ba<sub>3</sub>MgSi<sub>2</sub>O<sub>8</sub>, BaMgSiO<sub>4</sub> and SiO<sub>2</sub> were observed as impurity phases [12].

### Luminescence

The UV excited luminescence of Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup> at 295 K is observed as one broad band centred at 505 nm (figure 3), which corresponds to the earlier results [6, 13]. The emission in the green spectral region corresponds very well to the maximum sensitivity of the human eye and is thus very feasible to practical applications. The luminescence is due to the 4f<sup>6</sup>5d<sup>1</sup> → 4f<sup>7</sup> transition of the Eu<sup>2+</sup> ion. According to the structural studies [10], there is only one Ba<sup>2+</sup> site in the monoclinic Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub> lattice. The Eu<sup>2+</sup> ion is expected to occupy only this low symmetry (C<sub>1</sub>) Ba<sup>2+</sup> site. At 295 K, there is no indication of Eu<sup>2+</sup> occupying the Mg<sup>2+</sup> site. The R<sup>3+</sup> co-doping changes neither the luminescence wavelength nor the shape of the spectrum. The R<sup>3+</sup> ions may occupy either the Ba<sup>2+</sup> or Mg<sup>2+</sup> sites, since the ionic radii of the R<sup>3+</sup> ions is smaller than that of the Eu<sup>2+</sup> ion. However, probably the Ba<sup>2+</sup> site is preferred by the R<sup>3+</sup> ions because the compact four-fold coordination of the Mg<sup>2+</sup> ions with short Mg-O distance offers too small a site for the R<sup>3+</sup> ions. No luminescence from the co-dopants is observed, which indicates that neither the direct excitation nor the energy transfer from the Eu<sup>2+</sup> ion to the R<sup>3+</sup> ion occurs. However, the R<sup>3+</sup> luminescence from the 4f → 4f transitions can be masked by the much stronger luminescence due to the allowed 4f<sup>6</sup>5d<sup>1</sup> → 4f<sup>7</sup> transition of the Eu<sup>2+</sup> ion.

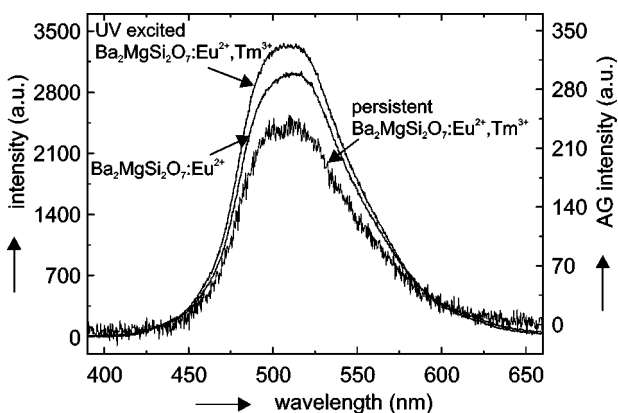


Figure 3. UV excited ( $\lambda_{exc} = 350$  nm) and persistent luminescence spectra of Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup> and Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup>,Tm<sup>3+</sup> at 295 K.

The excitation spectrum of Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup>,R<sup>3+</sup> at 295 K consists of at least four bands at 265, 315, 350 and 400 nm (figure 4), which agrees with the earlier results [13]. The excitation spectrum is due to the 4f<sup>7</sup> → 4f<sup>6</sup>5d<sup>1</sup> transitions of the Eu<sup>2+</sup> ion. No clear fine structure of the excitation bands due to the <sup>7</sup>F<sub>0-6</sub> levels is observed at 295 K. The co-doping with R<sup>3+</sup> ions does not change the excitation spectrum.

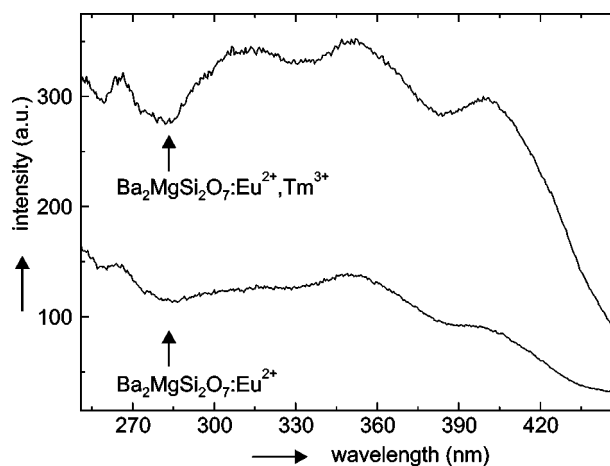


Figure 4. Excitation spectra ( $\lambda_{em} = 505$  nm) of Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup> and Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup>,Tm<sup>3+</sup> at 295 K.

### Persistent luminescence

The persistent luminescence spectrum of Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup> is identical to the UV excited one (figure 3). It can be concluded that the emitting centre is the same Eu<sup>2+</sup> ion in both luminescence phenomena. Most of the R<sup>3+</sup> ions enhance the persistent luminescence (table 1), but the Tm<sup>3+</sup> co-doping has much superior an effect on the persistent luminescence intensity and lifetime than any other R<sup>3+</sup> ion. This is a new result, since only the Nd<sup>3+</sup> and Dy<sup>3+</sup> ions have earlier been studied as co-dopants for the disilicates [5-8].

Table 1. Effect of the R<sup>3+</sup> co-doping on the persistent luminescence of Ba<sub>2</sub>MgSi<sub>2</sub>O<sub>7</sub>:Eu<sup>2+</sup>,R<sup>3+</sup> ( $\lambda_{exc} = 250-440$  nm).

Effect on persistent luminescence intensity	R <sup>3+</sup> ion
Enormous enhancement	Tm <sup>3+</sup>
Strong enhancement	Pr <sup>3+</sup> , Ho <sup>3+</sup> , Er <sup>3+</sup>
Enhancement	Ce <sup>3+</sup> , Sm <sup>3+</sup> , Gd <sup>3+</sup> , Tb <sup>3+</sup> , Dy <sup>3+</sup> , Yb <sup>3+</sup> , Lu <sup>3+</sup>
None	La <sup>3+</sup>
Suppression	Nd <sup>3+</sup>

The decay curve of the persistent luminescence of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Tm}^{3+}$  reveals of at least two processes, a rapid one prior to 20 min and a very slow one further on up to several hours (figure 5).

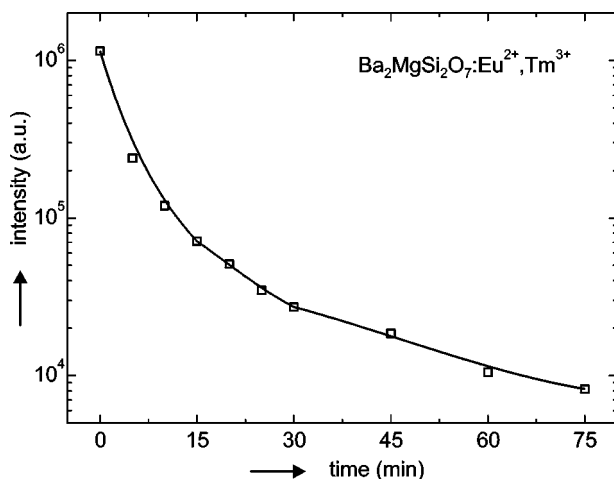


Figure 5. Persistent luminescence decay curve of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+},\text{Tm}^{3+}$ .

It is worth to notice that the  $\text{Dy}^{3+}$  and  $\text{Nd}^{3+}$  ions, which have been found to be superior to enhance the persistent luminescence of  $\text{Eu}^{2+}$  doped alkaline earth aluminates ( $\text{MAl}_2\text{O}_4:\text{Eu}^{2+}$ ;  $\text{M} = \text{Ca}, \text{Sr}$ ) [14], do not significantly enhance the persistent luminescence of the disilicates. In fact, the  $\text{Nd}^{3+}$  ion is the only effective quencher of the persistent luminescence of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$ . The  $\text{Tm}^{3+}$  co-doping has also been observed to enhance effectively the persistent luminescence of the  $\text{Eu}^{2+}$  doped calcium aluminate, however [14]. The role of the  $\text{R}^{3+}$  ion co-doping in the persistent luminescence materials is still uncertain. In  $\text{MAl}_2\text{O}_4:\text{Eu}^{2+}$ , the  $\text{R}^{3+}$  ions are proposed to act as traps [4] or at least modify them [14]. The chemical consequence of the  $\text{R}^{3+}$  co-doping is the creation of lattice defects by charge compensation, i.e. alkaline earth vacancies ( $V_M$ ) are then formed. These vacancies have been proposed to be essential to the persistent luminescence. In the case of the aluminates, the creation of the lattice defects alone does not, however, explain the phenomenon, since only some of the  $\text{R}^{3+}$  ions enhance the persistent luminescence. In the case of the disilicates, most of the  $\text{R}^{3+}$  ions enhance the persistent luminescence. Thus the crucial point for the enhancement of the persistent luminescence seems to be the creation of these charge compensation lattice defects e.g. barium (or magnesium) vacancies. The barium vacancies ( $V_{\text{Ba}}$ ) are already formed by the  $\text{Eu}^{2+}$  doping since a small amount of  $\text{Eu}^{3+}$  is always present in the samples. Also the oxygen vacancies ( $V_{\text{O}}$ ) are probably formed due to the reducing preparation conditions. The reasons making  $\text{Tm}^{3+}$  the most superior enhancer are still uncertain and more work is needed to reveal all the details.

## Thermoluminescence

Two TL peaks at 90 and 130°C are observed in the glow curve of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$  (figure 6). These peaks correspond to shallow and deep traps, respectively. The TL peak positions remain the same with increasing irradiation time. This kind of dose behaviour suggests that the TL peaks follow the first order kinetics i.e. no re-trapping takes place [15]. The TL peak positions are nearly ideal for the room temperature persistent luminescence [14]. One of the most effective persistent luminescence materials,  $\text{CaAl}_2\text{O}_4:\text{Eu}^{2+},\text{Nd}^{3+}$ , has two overlapping TL peaks at ca. 100 and 150°C, measured with the same set-up as used in this work [14]. In the  $\text{Eu}^{2+}$  doped calcium aluminates, the persistent (and thermo-) luminescence is due to lattice defects i.e. oxygen (calcium) vacancies, which create trapping levels for electrons (holes). Trapping is followed by thermal bleaching and energy transfer to the  $\text{Eu}^{2+}$  ion from an electron-hole recombination process [14]. A similar mechanism is also possible for the  $\text{Eu}^{2+}$  doped disilicates. The details of the mechanism(s) need, however, more experimental work.

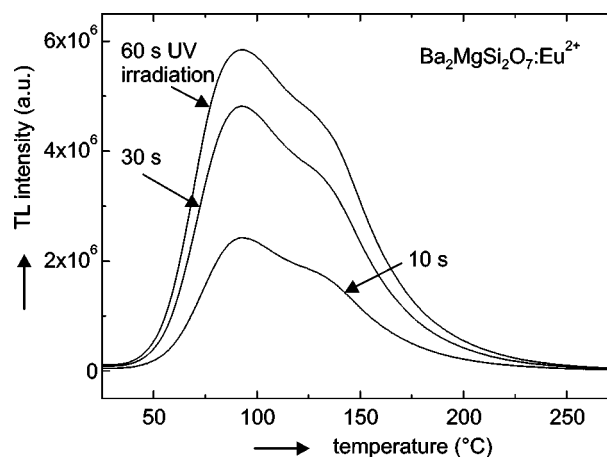


Figure 6. Thermoluminescence glow curve of  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$  (irradiation time: 10, 30, 60 s; delay time: 3 min; heating rate: 5°C/s).

## CONCLUSIONS

The new persistent luminescence material, monoclinic  $\text{Ba}_2\text{MgSi}_2\text{O}_7:\text{Eu}^{2+}$ , was successfully prepared by a solid state reaction with no or low amount of impurities. The UV excited and persistent luminescence were observed at the green region centred at 505 nm. Both luminescence phenomena are due to  $\text{Eu}^{2+}$  ions occupying the same single  $\text{Ba}^{2+}$  site in the lattice. In contrast to the  $\text{Eu}^{2+}$  doped alkaline earth aluminates, the creation of the lattice defects by the  $\text{R}^{3+}$  co-doping seems to be the main contribution to the enhancement of the persistent

luminescence, since almost all the  $R^{3+}$  ions enhance the persistent luminescence of  $Ba_2MgSi_2O_7:Eu^{2+}$ . Especially, the  $Tm^{3+}$  co-doping, which has been overlooked in the previous studies [6], enhances the persistent luminescence very strongly. Two thermoluminescence peaks at 90 and 130°C were observed in the glow curve of  $Ba_2MgSi_2O_7:Eu^{2+}$ , which is close to ideal for a room temperature persistent luminescence material. The persistent luminescence is probably due to lattice defects *i.e.* oxygen (alkaline earth vacancies), which can trap electrons (holes). The stored excitation energy is gradually released and transferred to the  $Eu^{2+}$  ion via an electron-hole recombination process. The details of the mechanism(s) need, however, more experimental work.

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#### LUMINESCENČNÍ VLASTNOSTI DIKŘEMIČITANU DIBARNATOHOŘEČNATÉHO DOPOVANÉHO $Eu^{2+}$ , $Ba_2MgSi_2O_7:Eu^{2+}$

T. AITASALO, J. HÖLSÄ, T. LAAMANEN, M. LASTUSAARI, L. LEHTO, J. NIITTYKOSKI, F. PELLÉ\*

University of Turku, Department of Chemistry, Laboratory of Inorganic Chemistry, FI-20014 Turku, Finland  
Graduate School of Materials Research, Turku, Finland  
\*CNRS, UMR 7574, ENSCP, Laboratoire de Chimie Appliquée de l'Etat Solide,  
11, r. P. et M. Curie, F-75231 Paris cedex 05, France

Nový materiál s persistentní luminescencí, monoklinický  $Ba_2MgSi_2O_7:Eu^{2+}$ , byl připraven reakcí v tuhé fázi. Luminescence jak excitovaná UV tak persistentní byla pozorována v zelené oblasti kolem 505 nm. Oba luminescenční jevy jsou umožněny stejným  $Eu^{2+}$  iontem obsazujícím jedinou pozici  $Ba^{2+}$  v mřížce. Společné dopování  $R^{3+}$  (kde R je lantanoid) obvykle zvyšuje persistentní luminescenci  $Ba_2MgSi_2O_7:Eu^{2+}$ . Obzvlášť ionty  $Tm^{3+}$  mají velmi značný vliv. Na obalové křivce termoluminescence  $Ba_2MgSi_2O_7:Eu^{2+}$  jsou patrné dva píky při 90 a 130°C, které jsou prakticky ideální pro materiály s persistentní luminescencí. Tato persistentní luminescence je patrně způsobena mřížkovými defekty, tj. kyslíkovými (kationtovými) vakancemi, které tvoří záchytové hladiny pro elektrony (díry). Rekombinace párů elektron-díra a přenosem energie na iont  $Eu^{2+}$  ion je pravděpodobným mechanismem persistentní luminescence. Podrobný mechanismus (mechanismy) by ale vyžadovalo provedení další experimentální práce. Stabilní dikřemičitany jsou velmi dobrými možnými materiály s persistentní luminescencí jako náhrady za hlinitany alkalických kovů dopované  $Eu^{2+}$ .