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Mid-infrared emission properties of erbium-1 doped fluorite-type crystals 2

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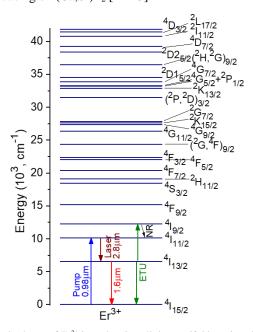
13 Abstract: We report on a comparative study of the spectroscopic properties and mid-infrared laser performance of five 5 at.% Er³⁺-doped fluorite-type crystals MF₂, including parent 14 15 compounds CaF₂, SrF₂, BaF₂, and solid-solution ("mixed") ones (Ca,Sr)F₂ and (Sr,Ba)F₂. In 16 the $M = Ca \rightarrow Sr \rightarrow Ba$ series, the host matrix phonon energy decreases, the absorption and midinfrared emission spectra of Er³⁺ become narrower and more structured, and the luminescence 17 lifetimes of the ${}^{4}I_{11/2}$ and ${}^{4}I_{13/2}$ Er³⁺ manifolds increase. The Er³⁺ transition probabilities were 18 calculated using the Judd-Ofelt theory. In the "mixed" compounds, the Er³⁺ ions tend to reside 19 20 in the larger / heavier cation environment. The low-temperature (12 K) spectroscopy evidences 21 the presence of a single type of clusters at this doping level; the crystal-field splitting for Er^{3+} 22 ions in clusters was determined. Continuous-wave low-threshold laser operation at $\sim 2.8 \ \mu m$ (the 23 ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2}$ transition) was achieved with all five $Er^{3+}:MF_2$ crystals. The maximum achieved laser slope efficiency was 37.9% (Er³⁺:CaF₂), 23.5% (Er³⁺:SrF₂) and 17.2% (Er³⁺:BaF₂). 24

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27 1. Introduction

28 Calcium fluoride (CaF₂, also known as fluorite in the mineral form) is a well-known laser host material for doping with trivalent rare-earth ions (RE³⁺) [1-3]. Undoped CaF₂ features good 29 30 thermal properties (high thermal conductivity and isotropic thermal expansion), low phonon 31 energy, low refractive index and broadband transparency. It also exhibits a unique tendency for strong RE^{3+} ion clustering even at moderate doping concentrations (>0.1 at.%), leading to 32 inhomogeneously broadened spectral bands [4-6]. As a result, the absorption and emission 33 34 spectra of RE^{3+} ions in CaF₂ greatly resemble those in fluoride glasses being almost structureless 35 and very broad. Such a "glassy-like" spectroscopic behavior is very appealing for broadband 36 wavelength tuning [7,8] and generation of ultrashort pulses in mode-locked lasers [9,10]. The 37 energy-transfer processes among the neighboring RE³⁺ ions (nonradiative energy-transfer, cross-38 relaxation, and energy-transfer upconversion) are greatly promoted in clusters [11]. This can be 39 used for boosting the efficiency of certain laser transitions of RE^{3+} ions. CaF_2 is a low-melting-40 point compound. Its growth is well-developed, e.g., by the Czochralski or Bridgman-41 Stockbarger methods.

42 CaF_2 belongs to the family of divalent metal fluorides, MF₂ (where M = Ca, Sr, Ba, Cd, or 43 Pb) [12-15]. These materials all belong to the cubic class (sp. gr. Fm3⁻m, fluorite-type structure). 44 The M^{2+} and F^{-} are located at face-centered cubic lattice points and tetrahedral voids, 45 respectively. Compared to CaF_2 , other MF₂ crystals are less studied for RE^{3+} doping but they are 46 also attractive as laser host media as they benefit from either a lower melting point, or better thermal properties, or lower phonon energies. Fluorite-type crystals can also form substitutional solid-solutions $(M1_{1-x}M2_x)F_2$ for the entire range of 0 < x < 1 [16-18]. For such "mixed" compositions, the melting point is expected to decrease further as compared to the parent compounds [19,20]. An additional spectral broadening is also expected due to the compositional disorder. The growth and laser operation of some RE³⁺-doped "mixed" fluorite-type crystals were reported mainly focusing on (Ca,Sr)F₂ [21-23].



53

54Fig. 1. Energy55spectra of Er³

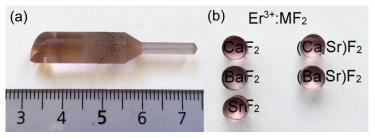
Fig. 1. Energy level scheme of Er^{3+} ions showing all the manifolds assigned in the absorption spectra of Er^{3+} :MF₂ crystals, pump and laser transitions, ETU – energy-transfer upconversion.

56 Erbium ions (Er^{3+}) are of interest for generation of mid-infrared radiation at ~2.8 µm [24,25] 57 according to the ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2}$ transition, Fig. 1. The low-phonon-energy behavior of MF₂ crystals 58 and the tendency for strong ion clustering promoting the energy-transfer upconversion stimulate 59 the interest in the development of mid-infrared $Er^{3+}:MF_2$ lasers. Labbe *et al.* first reported on a 60 mid-infrared 5 at.% Er³⁺:CaF₂ laser delivering 80 mW at 2.80 µm with a slope efficiency of 30% 61 and a small laser threshold of 23 mW [1]. Basyrova et al. demonstrated power scaling of a similar 62 laser generating 0.83 W at 2.80 µm with a slightly higher slope efficiency of 31.6% [26]. In 63 these studies, high-brightness laser pumping was implemented. Further power scaling was achieved using commercial InGaAs diode lasers as pump sources. Zong et al. developed a 64 diode-pumped 1.7 at.% Er³⁺:CaF₂ laser generating 2.32 W at 2.76 um at the expense of a lower 65 slope efficiency of 21.2% [27]. So far, Er³⁺:CaF₂ [26,27], Er³⁺:SrF₂ [28] and Er³⁺:(Ca,Sr)F₂ [23] 66 crystals have been studied for mid-infrared lasers. Note that Er³⁺:CaF₂ can also be obtained in 67 68 the form of transparent ceramics. Sulc *et al.* reported on a pulsed diode-pumped 5 at.% Er^{3+} :CaF₂ 69 ceramic laser with a broad tuning range of 2687 - 2805 nm (118 nm) [29].

70 Despite the existence of multiple studies for several $Er^{3+}:MF_2$ crystal compositions, their 71 spectroscopic and mid-infrared laser properties have not been directly compared so far. In the 72 present work, we report on a comparative study of mid-infrared emission properties of five 73 fluorite-type Er^{3+} -doped MF₂ crystals, including parent and solid-solution compounds.

74 2. Crystal growth

The MF₂ crystals (M = Ca, Sr, Ba) melt congruently at relatively low temperatures (cf. Table 1) and they can be grown by the Bridgman-Stockbarger or Czochralski methods. The $Er^{3+}:MF_2$ crystals were grown by the Bridgman method using graphite crucibles (Φ 7-8 mm, height: 40 78 mm). The MF₂ (M = Ca, Sr, Ba) powders (purity: 4N, Sigma-Aldrich) and ErF₃ powder obtained 79 by fluorination of the Er_2O_3 precursor (4N, Alfa Aesar). Five compositions were tested: M = Ca, 80 Sr, Ba, $Ca_{0.5}Sr_{0.5}$ and $Sr_{0.5}Ba_{0.5}$. The doping level was 5 at.% Er^{3+} (with respect to M^{2+} cations). The optical quality and spectroscopic properties of RE^{3+} -doped MF₂ crystals are sensitive to 81 82 even small pollution of oxygen / water in the growth chamber as they can lead to the presence 83 of oxygen-assisted sites for the dopant ions or even formation of a translucent oxyfluoride phase. 84 To avoid that, the growth chamber was sealed to vacuum ($<10^{-5}$ mbar) and refilled with a mixture 85 of $Ar + CF_4$ gases. The starting reagents were well mixed and placed into the crucible which was 86 then heated slightly above (~30-50 °C) the melting point and the solution was homogenized for 87 3-4 hours (h). The growth was ensured by a vertical translation of the crucible in a vertical 88 temperature gradient of 30-40 °C/cm. After the growth was completed, the crystals were cooled 89 down to room temperature (20 °C) within 48 h.



90 91 92

Fig. 2. Photographs of Er^{3+} :MF₂ crystals: (a) an as-grown Er^{3+} :CaF₂ crystal boule; (b) cut and polished Er^{3+} :MF₂ samples.

For the "mixed" crystals, the melting point is reduced as compared to those of the parent compounds. E.g., for $Er^{3+}:(Ca,Sr)F_2$ and $Er^{3+}:(Sr,Ba)F_2$, it is $T_f = 1373$ °C and 1315 °C, respectively (compare with 1477 °C, 1418 °C and 1386 °C for SrF_2 , CaF_2 and BaF_2 , respectively).

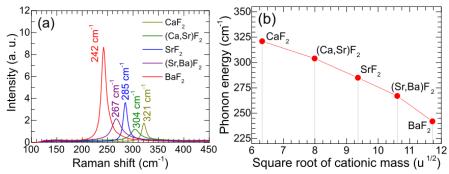
97 The as-grown $Er^{3+}:MF_2$ crystals with a cylindrical shape (Φ 7-8 mm, length: 35 - 40 mm) 98 were transparent and rose-colored due to the Er^{3+} doping, Fig. 2(a). Samples for spectroscopic 99 and laser studies were cut from the central part of the cylindrical barrels with a thickness of 6-7 100 mm and then polished to laser-grade quality, Fig. 2(b).

101 3. Raman spectra

102 The Raman spectra of Er^{3+} :MF₂ crystals, Fig. 3(a), were measured using a confocal microscope 103 (InVia, Renishaw) equipped with a ×50 Leica objective and an Ar⁺ ion laser (457 nm). Fluorite-104 type crystals have O_h symmetry and a triatomic unit cell thus exhibiting only one Raman-active 105 mode at the center of the Brillouin zone having a T_{2g} symmetry [30]. Indeed, the Raman spectra of all the studied Er^{3+} :MF₂ crystals contain a single intense peak assigned to this vibration. 106 107 Frequently, MF₂ crystals may exhibit additional broad Raman bands in the spectral range of 100 108 -600 cm^{-1} owing to structure defects (interstitial / vacant anion sites) [30]. Such a behavior is 109 not observed in our crystals.

For Er³⁺:CaF₂, Er³⁺:SrF₂ and Er³⁺:BaF₂ crystals, the peak frequency of the Raman mode and 110 111 its linewidth (FWHM) are 321 / 11.0 cm⁻¹, 285 / 10.2 cm⁻¹ and 242 / 12.0 cm⁻¹, respectively. 112 Thus, the latter compound is the most favorable one in terms of low-phonon-energy behavior. 113 For the "mixed" compositions, the Raman peak broadens and is reduced in intensity and the peak 114 position takes an intermediate place between those for the corresponding parent compounds, 115 indicating an even distribution of the host-forming cations throughout the structure (a formation 116 of a substitutional solid-solution) [32]. E.g., for $Er^{3+}(Ca,Sr)F_2$, the peak Raman frequency is 304 cm⁻¹ and the peak linewidth is 25.9 cm⁻¹. 117

118 The phonon energy of Er^{3+} :MF₂ decreased monotonically with increasing the cationic mass 119 in agreement with the classical approach, $v = (1/2\pi)(k/\mu)^{1/2}$, where *k* is the force constant and μ 120 is the reduced mass of the M – F system [33].



121

122 123 **Fig. 3.** Raman spectroscopy of Er^{3+} :**M**F₂ crystals: (a) Raman spectra, $\lambda_{\text{exc}} = 457 \text{ cm}^{-1}$, *numbers* – peak frequencies; (b) phonon energy vs. the square root of the average cationic mass.

124 4. Optical spectroscopy

125 4.1 Optical absorption

The absorption spectra of Er³⁺ ions were measured using a spectrophotometer (Lambda 1050, 126 Perkin Elmer). They are shown in Fig. 4. Here, the assignment of Er^{3+} transitions is according 127 128 to Carnall *et al.* [34]. The absorption spectra for both parent and "mixed" Er³⁺:MF₂ crystals are 129 smooth and broad owing to inhomogeneous spectral broadening caused by a strong ion clustering. In the series $M = Ca \rightarrow Sr \rightarrow Ba$, the complexity and diversity of RE^{3+} ion clusters 130 131 in MF₂ crystals decrease leading to more intense and structured absorption bands which also exhibit a slight blue-shift [12]. Indeed, for the ${}^{4}I_{15/2} \rightarrow {}^{4}I_{11/2}$ transition, which is used for pumping 132 mid-infrared erbium lasers, the peak absorption cross-section, σ_{abs} , varies from 2.77×10⁻²¹ cm² 133 at 972.3 nm (Er³⁺:BaF₂) to 2.59×10⁻²¹ cm² at 969.5 nm (Er³⁺:SrF₂), to 2.22×10⁻²¹ cm² at 967.6 134 135 nm (Er³⁺:CaF₂), while the corresponding absorption bandwidth is 12.9 nm (Er³⁺:BaF₂), 16.9 nm (Er³⁺:SrF₂), and 22.2 nm (Er³⁺:CaF₂). 136

A close look at the absorption spectra of "mixed" crystals indicate that there is a great 137 similarity between those of $(Er^{3+};(Ca,Sr)F_2 \text{ and } Er^{3+};SrF_2)$ and $(Er^{3+};(Sr,Ba)F_2 \text{ and } Er^{3+};BaF_2)$ 138 139 ones, suggesting that the dopant ions in such solid-solution compounds tend to reside in clusters 140 with a local surrounding predominantly composed of one of the two host-forming cations (namely, the heavier / larger one – Sr^{2+} or Ba^{2+} , respectively). This suggests that Er^{3+} clusters 141 142 have a tendency to sit in the heavier-cation environment within the solid-solution $(M_{1-x}M_{2x})F_2$ 143 crystals. A similar behavior was observed previously for clusters of Nd³⁺/Lu³⁺ ions in "mixed" 144 (Sr,Ba)F₂ crystals [35].

145 4.2 Judd-Ofelt analysis

146 The measured absorption spectra of Er^{3+} ions in the five studied MF_2 crystals were used to 147 calculate the transition probabilities by means of the standard Judd-Ofelt (J-O) theory [36,37]. 148 The reduced squared matrix elements $U^{(k)}$ (k = 2, 4, 6) were calculated using the free-ion 149 parameters from [38]. The magnetic dipole (MD) contributions to transition intensities (for 150 $\Delta J = J - J' = 0, \pm 1$) were calculated within the Russell–Saunders approximation using Er^{3+} wave 151 functions under the free-ion assumption.

152 Table 1 summarizes the experimental and calculated absorption oscillator strengths (f_{exp} and 153 f_{calc} , respectively) for the three parent compounds, Er^{3+} :CaF₂, Er^{3+} :SrF₂ and Er^{3+} :BaF₂. There 154 exists a direct relation between the absorption oscillator strength / integrated absorption cross-155 section and the radiative lifetime of the excited-state (the principle of reciprocity, referring to Einstein coefficients). For Er^{3+} transitions from the ground-state (${}^{4}I_{15/2}$) to the two lower-lying 156 excited-states (${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$), the f_{calc} value decreases in the M = Ca \rightarrow Sr \rightarrow Ba series, so that 157 158 an opposite tendency is expected for the radiative lifetimes of these two states. The root mean 159 square (r.m.s.) deviation between the f_{exp} and f_{calc} values is relatively low for all the tested 160 Er^{3+} :MF₂ crystals, lying in the range of 0.137 – 0.257.

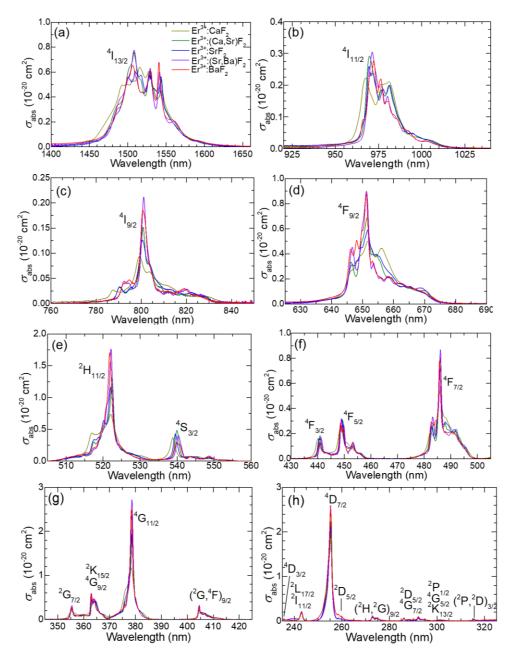




Fig. 4. (a-h) Absorption spectra of Er^{3+} ions in MF₂ crystals.

The J-O (intensity) parameters Ω_2 , Ω_4 , Ω_6 for Er³⁺ ions in MF₂ crystals are listed in Table 2. 163 164 The determined J-O parameters were used to calculate the probabilities of spontaneous 165 radiative transitions of Er³⁺ ions. In Table 3, we list the parameters relevant for mid-infrared 166 laser operation, i.e., the radiative lifetimes τ_{rad} of the ${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$ states and the luminescence branching ratio $\beta_{JJ'}$ for the ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2}$ transition. As expected, in the M = Ca \rightarrow Sr \rightarrow Ba 167 168 series, the τ_{rad} values for the considered excited-states tend to increase from 7.09 / 6.53 ms 169 $(Er^{3+}:CaF_2)$ to 7.57 / 6.99 ms $(Er^{3+}:SrF_2)$ and further to 7.52 / 7.11 ms $(Er^{3+}:BaF_2)$. The considered $\beta_{JJ'}$ value is also higher for Sr²⁺ and Ba²⁺-containing crystals. 170

Transition	Er ³⁺ :CaF ₂		$Er^{3+}:SrF_2$		Er ³⁺ :BaF ₂	
${}^{4}I_{15/2} \rightarrow$	f_{exp} ,	$f_{\text{calc}}, \text{J-O}$	f_{exp} ,	$f_{\text{calc}}, \text{J-O}$	f _{exp} ,	$f_{\text{calc}}, \text{J-O}$
$^{2S+1}L_{I}$	×10 ⁻⁶	×10 ⁻⁶	×10 ⁻⁶	×10 ⁻⁶	×10 ⁻⁶	×10 ⁻⁶
⁴ I _{13/2}	2.496	$1.643^{ED} +$	2.129	$1.510^{ED} +$	2.220	$1.404^{ED} +$
		0.448^{MD}		0.448^{MD}		0.461 ^{MD}
⁴ I _{11/2}	0.714	0.693 ^{ED}	0.643	0.631 ^{ED}	0.598	0.592^{ED}
⁴ I _{9/2}	0.333	0.229 ^{ED}	0.328	0.283 ^{ED}	0.343	0.333 ^{ED}
4F _{9/2}	2.264	2.139 ^{ED}	2.217	2.283 ^{ED}	2.265	2.406^{ED}
${}^{4}S_{3/2} + {}^{2}H_{11/2}$	3.531	3.366 ^{ED}	3.422	3.544^{ED}	4.387	4.760 ^{ED}
4F7/2	2.141	2.454^{ED}	2.396	2.342^{ED}	2.205	2.236 ^{ED}
${}^{4}F_{5/2} + {}^{2}F_{3/2}$	1.266	1.300 ^{ED}	1.114	1.173 ^{ED}	0.962	1.056 ^{ED}
$^{2}G_{9/2}$	0.898	0.970^{ED}	0.637	0.902^{ED}	0.603	0.840^{ED}
${}^{4}G_{11/2} + {}^{2}K_{15/2} +$	7.888	$7.917^{ED} +$	8.585	$8.474^{ED} +$	11.400	11.168 ^{ED}
${}^{4}G_{9/2} + {}^{2}G_{7/2}$		0.053 ^{MD}		0.053 ^{MD}		$+ 0.055^{MD}$
r.m.s. dev.		0.217		0.137		0.234
$f_{\rm exp}$ and $f_{\rm calc}$ - experi-	imental and ca	lculated absorp	ption oscillato	r strengths, res	pectively, ED	 electric dipol

Table 1. Absorption Oscillator Strengths^a for Er³⁺ Ions in Parent MF₂ (M = Ca, Sr, Ba) Crystals

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Table 2. Judd-Ofelt Parameters of Er³⁺ lons in MF₂ Crystals

Host crystal	$\Omega_2, 10^{-20} \mathrm{cm}^2$	Ω_4 , 10 ⁻²⁰ cm ²	$\Omega_6, 10^{-20} \mathrm{cm}^2$
CaF ₂	1.436	1.364	1.892
(Ca,Sr)F ₂	1.244	1.483	1.720
SrF_2	1.477	1.701	1.701
(Sr,Ba)F2	1.519	1.836	1.529
BaF ₂	2.397	1.964	1.487

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Table 3. Selected Probabilities^a of Spontaneous Radiative Transitions of Er³⁺ in MF₂ crystals

Host crystal	$\tau_{\rm rad}({}^{4}{\rm I}_{13/2}),{ m ms}$	$\tau_{\rm rad}({}^{4}{\rm I}_{11/2}),{ m ms}$	$\beta_{\rm JJ'}({}^{4}{\rm I}_{11/2} \rightarrow {}^{4}{\rm I}_{13/2}), \%$
CaF ₂	7.09	6.53	14.9
(Ca,Sr)F ₂	7.64	7.06	15.8
SrF_2	7.57	6.99	16.0
(Sr,Ba)F ₂	7.90	7.56	16.5
BaF_2	7.52	7.11	16.1
$a\tau_{\rm rad}$ – radiative life	etime, $\beta_{JJ'}$ – luminesce	ence branching ratio.	

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177 4.3 Emission spectra and luminescence lifetimes

MD – magnetic dipole.

178 The luminescence spectra of Er^{3+} ions in the mid-infrared (the ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2}$ transition) were 179 measured using an optical spectrum analyzer (OSA, Yokogawa AQ6376) and a ZrF4 fiber. The excitation source was a Ti:Sapphire laser tuned to \sim 970 nm. The OSA was purged with N₂ gas. 180 181 To remove the effect of the residual water vapor absorption in air, the set-up was calibrated 182 using a 20 W quartz iodine lamp.

183 The stimulated-emission (SE) cross-sections, σ_{SE} , were calculated using the Füchtbauer-184 Ladenburg equation [39]:

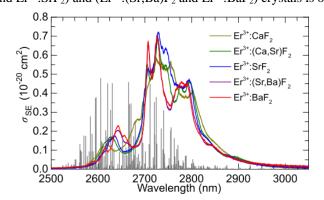
$$\sigma_{\rm SE}(\lambda) = \frac{\lambda^5}{8\pi < n >^2} \frac{B(JJ')W'(\lambda)}{\int \lambda W'(\lambda) d\lambda},$$
(1)

186 where λ is the light wavelength, $\langle n \rangle$ is the refractive index of the crystal at the mean emission wavelength, τ_{rad} corresponds to the ${}^{4}I_{11/2}$ state and $\beta_{JJ'}$ – to the ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2}$ transition (cf. 187 188 Table 3), c is the speed of light, and $W'(\lambda)$ is the measured luminescence spectrum corrected for 189 the response of the set-up.

190 The SE cross-section spectra for Er^{3+} ions in MF₂ crystals are shown in Fig. 5. Similarly to the absorption spectra, a profound inhomogeneous broadening is observed for both the parent 191 192 and "mixed" Er³⁺:MF₂ crystals owing to the rare earth ion clustering. For all the studied crystals, the emission spectra are very broad extending from 2.55 to 3.05 µm and the main emission peak 193 194 appears around 2.72 µm. Such a behavior is beneficial for broadly tunable and potentially

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195 mode-locked lasers. The spectra become more structured in the $M = Ca \rightarrow Sr \rightarrow Ba$ series. 196 Also for the solid-solution compounds, a great similarity between the emission spectra of 197 (Er³⁺:(Ca,Sr)F₂ and Er³⁺:SrF₂) and (Er³⁺:(Sr,Ba)F₂ and Er³⁺:BaF₂) crystals is observed.

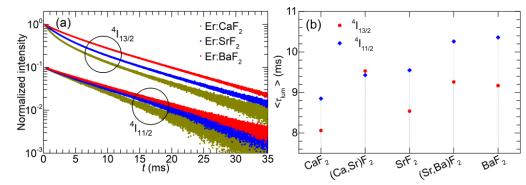


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199 Fig. 5. Stimulated-emission (SE) cross-sections, σ_{SE} , for the ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2}$ Er³⁺ transition in MF₂ **200 201** crystals, corrected for the structured water vapor absorption in air (in *grey*, arb. units, according to the HITRAN database).

The highest SE cross-section is observed for Er^{3+} :SrF₂, $\sigma_{\text{SE}} = 7.19 \times 10^{-21} \text{ cm}^2$ at 2729 nm and at longer wavelengths, two other intense and broad peaks appear ($\sigma_{\text{SE}} = 6.38 \times 10^{-21} \text{ cm}^2$ at 2745 nm and 4.69×10⁻²¹ cm² at 2794 nm).

Luminescence decays were studied under resonant excitation using a ns optical parametric 205 206 oscillator (Horizon, Continuum), a 1/4 m monochromator (Oriel 77200), an InGaAs detector 207 and an 8 GHz oscilloscope (DSA70804B, Tektronix). To reduce the reabsorption (radiation trapping) effect on the measured kinetics, the samples were finely ground into powders. The 208 measured luminescence decay curves from the ${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$ Er³⁺ states in the three parent 209 crystals, CaF₂, SrF₂ and BaF₂, are shown in Fig. 6(a). They deviate from the single-exponential 210 211 law (especially for ${}^{4}I_{13/2}$) owing to the strong ETU from these long-living states. Thus, the mean 212 luminescence lifetimes $\langle \tau_{lum} \rangle = \int t \cdot I(t) dt / \int I(t) dt$ were determined.



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Fig. 6. Luminescence dynamics from the ${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$ Er³⁺ manifolds in MF₂ crystals: (a) luminescence decay curves under resonant excitation of Er³⁺ ions in CaF₂, SrF₂, and BaF₂, $\lambda_{exc} = 1.48 \ \mu\text{m}$, $\lambda_{\text{lum}} = 1.57 \ \mu\text{m}$ (the ${}^{4}I_{13/2}$ state), $\lambda_{exc} = 0.97 \ \mu\text{m}$, $\lambda_{\text{lum}} = 1.01 \ \mu\text{m}$ (the ${}^{4}I_{11/2}$ state); (b) mean luminescence lifetimes $\langle \tau_{\text{lum}} \rangle$ as a function of the host composition.

218The summary of the $\langle \tau_{lum} \rangle$ values for the five studied $Er^{3+}:MF_2$ crystals is given in Fig. 6(b).219With increasing the average radius / atomic mass of the M^{2+} host-forming cations (in the M =220 $Ca \rightarrow Sr \rightarrow Ba$ series), and, accordingly, decreasing the phonon energy of the host matrix, both221the ${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$ luminescence lifetimes tend to increase, from 8.06 / 8.85 ms ($Er^{3+}:CaF_2$) to2228.54 / 9.55 ms ($Er^{3+}:SrF_2$) and further to 9.17 / 10.36 ms ($Er^{3+}:BaF_2$). This behavior agrees with223that for the calculated radiative lifetimes of these manifolds. The ratio of the upper-to-lower

224 laser level lifetimes is favorable for all the studied crystals being weakly dependent on the host 225 matrix composition. The long luminescence lifetime of the upper laser level for the mid-infrared 226 transition (${}^{4}I_{11/2}$) is a prerequisite for a low-threshold behavior.

227 Note that the measured luminescence lifetimes are slightly exceeding the radiative ones 228 calculated using the J-O theory (cf. Table 3). One possible reason for that is the residual 229 reabsorption effect within the Er^{3+} ion clusters.

Parameter / Crystal	CaF ₂	(Ca,Sr)F ₂	SrF ₂	(Sr,Ba)F ₂	BaF ₂
λ_{abs} , nm	967.6	969.7	969.5	971.4	972.3
$\sigma_{\rm abs}, 10^{-21} {\rm cm}^2$	2.22	2.91	2.59	3.04	2.77
$\lambda_{\rm em}, nm$	2724.8	2727.6	2728.9	2726.6	2726.5
$\sigma_{\rm SE}, 10^{-21} {\rm cm}^2$	5.87	6.90	7.22	7.11	6.92
$<\tau_{\text{lum}}>(^{4}\text{I}_{13/2}), \text{ms}$	8.06	9.53	8.54	9.26	9.17
$<\tau_{\rm lum}>({}^{4}{\rm I}_{11/2}),{ m ms}$	8.85	9.43	9.55	10.26	10.36

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Table 4. Spectroscopic Characteristics^a of Er³⁺:MF₂ Crystals

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 $a_{\lambda_{abs}}, \lambda_{em}$ – peak absorption / emission wavelengths, respectively, $\sigma_{abs}, \sigma_{SE}$ – peak absorption / SE cross-sections, respectively, $\langle \tau_{lum} \rangle$ - average luminescence lifetime.

233 *4.4 Low-temperature spectroscopy*

For low-temperature (LT, 12 K) absorption and luminescence studies, we have used an APD DE-202 closed-cycle cryo-cooler equipped with an APD HC 2 Helium vacuum cryo-compressor and a Laceshore 330 temperature controller. For absorption measurements, a 20 W quartz lamp with a calibrated spectral output was used. The spectra were measured using optical spectrum analyzers (Ando AQ6315A and Yokogawa AQ6375E). The luminescence was excited by a Ti:Sapphire laser tuned to ~800 nm.

The LT absorption and emission spectra are shown in Fig. 7 and Fig. 8, respectively. In each graph, we compare the spectrum of a "mixed" compound with those of both parent crystals. The LT absorption spectra were plotted versus the phonon energy giving access to the splitting of the ⁴I_{13/2} and ⁴I_{11/2} excited-states, while the LT emission spectra were plotted versus (E_{ZPL} – photon energy), where E_{ZPL} is the zero-phonon line (ZPL) energy giving access to the splitting of the ground-state ⁴I_{15/2}.

By analyzing the spectra, several conclusions can be derived:

247(i) The absorption and emission spectra of Er^{3+} ions in MF2 crystals contain very broad bands248even at 12 K indicating a significant inhomogeneous spectral broadening due to the rare-earth ion249clustering. The spectra become more structured in the series $M = Ca \rightarrow Sr \rightarrow Ba$ indicating250smaller variety of cluster geometries;

(ii) The spectra of "mixed" fluorite-type crystals exhibit additional broadening as compared to the corresponding parent compounds due to the presence of two different host-forming cations. The spectra of such "mixed" crystals are more similar to those of the heavier-cation parent (e.g., (Ca,Sr)F₂ and SrF₂, (Ba,Sr)F₂ and BaF₂). This corroborates the observation made in Section 4.3, confirming that the majority of Er^{3+} ions tend to reside in the vicinity of heavier cations within "mixed" crystals;

(iii) The total Stark splitting of Er^{3+} multiplets in clusters in MF₂ crystals decreases in the M = Ca \rightarrow Sr \rightarrow Ba series, and the corresponding barycenter energies experience a progressive red-shift. The strength of the crystal-field is expected to be larger for smaller sites (shorter M – F and M - M distances, in our case) due to the stronger lattice distortion on the dopant ion. Indeed, the lattice constant increases in the series CaF₂ (5.45 Å) \rightarrow SrF₂ (5.80 Å) \rightarrow BaF₂ (6.20 Å).

Based on the LT absorption and emission spectra, the crystal-field splitting of the ${}^{4}I_{15/2}$, ${}^{4}I_{13/2}$ and ${}^{4}I_{11/2}$ multiplets of Er^{3+} ions forming clusters in the three parent MF₂ crystals (M = Ca, Sr, Ba), was determined, Table 5. The experimental Stark splitting of the ${}^{4}I_{11/2}$ and ${}^{4}I_{13/2}$ multiplets relevant for the 2.8 µm laser operation is also compared in Fig. 9. In the previous studies on siteselective spectroscopy of Er^{3+} ions in CaF₂ crystals grown under oxygen-free atmosphere, multiple possible sites were identified [40-42]. At very low doping levels (<0.05 at.%), the Er^{3+}

ions are mostly isolated and are distributed over tetragonal (A, C_{4v}), trigonal (B, C_{3v}) and cubic (O_h) sites, depending on the relative position of the charge-compensating interstitial fluorine anion (F_i⁻), namely at the (1,0,0) positions, at the (1,1,1) positions or sufficiently far from the dopant ion to exert negligible perturbation, respectively [40]. For higher doping levels of >0.1 at.%, the dopant ions form clusters of several types (assigned as C-sites, being close to dimers with a distorted C_{3v} symmetry, and D(1) and D(2) sites corresponding to larger agglomerates of Er³⁺ -F_i⁻ pairs).

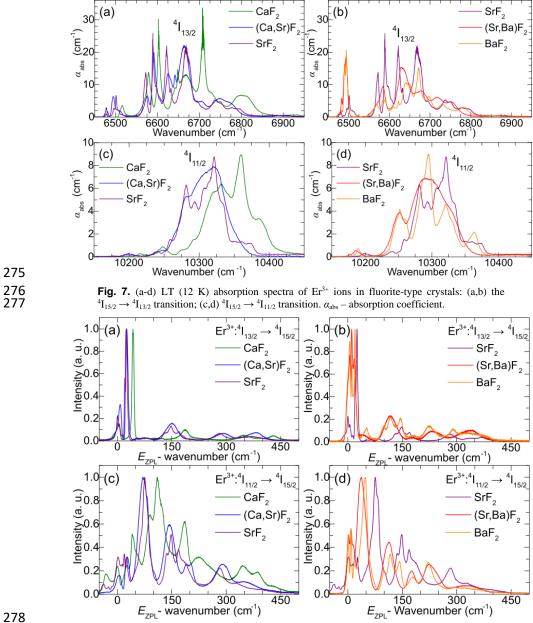




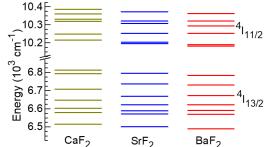
Fig. 8. (a-d) LT (12 K) luminescence spectra of Er^{3+} ions in fluorite-type crystals: (a,b) the ${}^{4}I_{132} \rightarrow {}^{4}I_{152}$ transition; (c,d) ${}^{4}I_{11/2} \rightarrow {}^{4}I_{15/2}$ transition. E_{ZPL} – zero-phonon-line energy.

281 For the studied heavily doped Er^{3+} :MF₂ crystals, we were not able to confirm the existence of 282 two significantly different groups of ion clusters (D(1) and D(2)), as the LT emission spectra were 283 almost independent on the excitation wavelength. Moreover, the bands in the LT spectra of ~5 284 at.% Er^{3+} -doped crystals (assigned to a single type of cluster D sites) experience an additional 285 broadening and spectral shifts as compared to those in 0.1 at.% Er³⁺-doped crystals (assigned to 286 D(1) and D(2) sites). Thus, we assumed that almost all the Er^{3+} ions form large-scale agglomerates (D) with relatively close spectroscopic properties. Previously, it was suggested that for all the 287 288 heavily doped MF₂ crystals (M = Ca, Sr, Ba) and their solid-solutions, such agglomerates most likely correspond to hexameric Y_6F_{37} superstructure units, which are nearly identical in volume 289 290 and shape to the Ca_2F_{32} building blocks of the fluorite lattice and, consequently, they can be 291 easily incorporated into this lattice while accommodating the excess Fi⁻ anions [5,43]. The local 292 crystal-field symmetry for the dopant ions in Y_6F_{37} clusters is tetragonal (C_{4v}) [5].

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Table 5. Crystal-Field Splitting of Selected Er³⁺ Multiplets in CaF₂, SrF₂, and BaF₂

Crystal	Er ³⁺	Sub-leve	el / Energy	(cm ⁻¹)					
-	$^{2S+1}L_J$	1	2	3	4	5	6	7	8
CaF ₂	${}^{4}I_{15/2}$	0	42	90	110	186	228	346	387
	${}^{4}I_{13/2}$	6516	6579	6602	6648	6708	6793	6812	
	${}^{4}I_{11/2}$	10215	10248	10320	10331	10360	10386		
SrF_2	${}^{4}I_{15/2}$	0	25	63	75	148	187	283	330
	${}^{4}I_{13/2}$	6501	6572	6589	6622	6669	6737	6796	
	${}^{4}I_{11/2}$	10196	10204	10282	10307	10321	10373		
BaF_2	${}^{4}I_{15/2}$	0	18	34	48	117	142	222	293
	${}^{4}I_{13/2}$	6489	6569	6590	6622	6672	6730	6784	
	${}^{4}I_{11/2}$	10182	10190	10252	10295	10321	10363		



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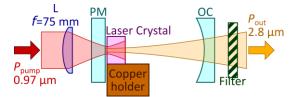
Fig. 9. Experimental Stark splitting of the ${}^{4}I_{11/2}$ and ${}^{4}I_{13/2}$ multiplets of Er^{3+} ions forming clusters in heavily doped $Er^{3+}:MF_{2}$ crystals.

The analysis of Table 2 confirms a decreased total Stark splitting of the multiplets and a redshift of the zero-phonon line for Er^{3+} ions in the $M = Ca \rightarrow Sr \rightarrow Ba$ series.

299 5. Laser operation

300 5.1 Laser setup

301 The scheme of the laser set-up is shown in Fig. 10. Cylindrical samples with a thickness of 6.53-302 6.99 mm and a diameter of ~7 mm were cut from the central parts of the as-grown $Er^{3+}:MF_2$ 303 crystal boules. They were polished to laser-grade quality with good parallelism (<5) from both 304 sides and left uncoated. The laser elements were mounted on a passively cooled Cu-holder using 305 a silver paint for better heat removal. A hemispherical cavity was implemented. It was formed by 306 a flat pump mirror (PM) coated for high transmission (HT, T = 85.7%) at 0.97 µm and high 307 reflection (HR) at $2.6 - 3.0 \mu m$, and a set of concave (radius of curvature: RoC = -100 mm) output couplers (OC) having a transmission T_{OC} in the range of 0.33% - 4% at 2.7 - 2.9 μ m. 308 309 The crystal was placed near the PM at a small distance (<1 mm). The geometrical cavity length 310 was ~99 mm. The pump source was a CW Ti:Sapphire laser delivering up to 3.2 W at 0.97 µm 311(addressing the ${}^{4}I_{15/2} \rightarrow {}^{4}I_{11/2}$ Er³⁺ absorption peak) with a diffraction-limited beam quality (M2312 \approx 1). The pump radiation was focused into the laser crystal through the PM using an313antireflection-coated achromatic lens (focal length: f = 75 mm) resulting in a pump spot size of314 $2w_P = 66 \pm 5 \ \mu m$. The pumping was in single pass. The residual (non-absorbed) pump after the315OC was filtered out using a long-pass filter (Spectrogon, LP1400). The laser spectra were316measured using a ZrF4 fiber (Thorlabs) and a spectrum analyzer (Bristol, 771 series). The laser317mode profile in the far-field was captured using a camera (Pyrocam IIIHR, Ophir-Spiricon).



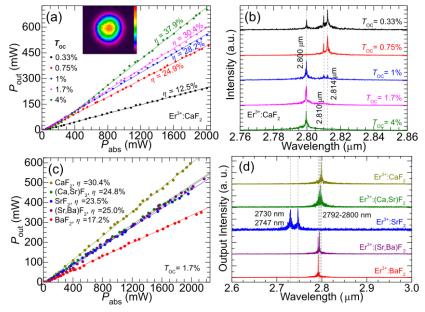
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Fig. 10. Schematic of the laser setup: L – aspherical focusing lens; PM – flat pump mirror; OC

 320 – curved output coupler.

321 5.2 Laser performance

CW mid-infrared laser operation was obtained with all five studied Er³⁺:MF₂ crystals. The best 322 323 laser performance was achieved with the Er^{3+} :CaF₂ crystal: an output power of 702 mW was 324 extracted at 2800 nm with a slope efficiency η of 37.9% (vs. the absorbed pump power) when 325 using the output coupler with $T_{\rm OC} = 4\%$, Fig. 11(a). With increasing output coupling from 0.33% to 4%, the laser threshold gradually increased from 16 mW to 60 mW. For Er³⁺:CaF₂, the 326 327 measured pump absorption reached 81.9%. The optical-to-optical efficiency (vs. the pump power 328 incident on the crystal) η_{opt} was 31.0%. The output dependences were linear within the studied 329 range of pump powers. Further power scaling was limited by the available pump. The achieved laser slope efficiency is slightly higher than the Stokes limit, $\eta_{St,L} = \lambda_P / \lambda_L = 34.6\%$, indicating the 330 role of the ETU process ${}^{4}I_{13/2} + {}^{4}I_{13/2} \rightarrow {}^{4}I_{9/2} + {}^{4}I_{15/2}$, cf. Fig. 1, refilling the upper laser level and 331 332 depopulating the intermediate ⁴I_{13/2} state.



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Fig. 11. Mid-infrared $\text{Er}^{3+}:\text{MF}_2$ lasers: (a,b) $\text{Er}^{3+}:\text{CaF}_2$ laser: (a) input-output dependences, η – slope efficiency, *inset* – far-field mode profile, $P_{abs} \sim 1.5$ W, $T_{OC} = 1.7\%$; (b) typical laser spectra; (c,d) a comparison of (c) power transfer characteristics and (d) laser spectra for five $\text{Er}^{3+}:\text{MF}_2$ crystals, $T_{OC} = 1.7\%$.

338 The typical emission spectra of the Er^{3+} :CaF₂ laser are shown in Fig. 11(b), measured well 339 above the laser threshold. For small output coupling (<1%), laser emission at 2810 and 2814 nm 340 was observed and for higher $T_{\rm OC}$, the laser operated at 2800 nm. These wavelengths correspond to the long-wave emission peak of Er^{3+} ions in CaF_2 and match the transparency ranges between 341 342 the structured water vapor absorption lines (cf. Fig. 5). Note that due to strong resonant excited-343 state absorption from the terminal laser level with a non-negligible population, ${}^{4}I_{13/2} \rightarrow {}^{4}I_{11/2}$, causing reabsorption of the laser photons, the ${}^{4}I_{11/2} \rightarrow {}^{4}I_{13/2} \text{ Er}^{3+}$ laser transition represents a 344 345 quasi-three-level laser scheme with reabsorption, which explains the blue-shift of the laser 346 spectra with increasing the output coupling.

347 The Er^{3+} :CaF₂ laser operated on the fundamental transverse mode, as confirmed by the 348 measured $M^2 < 1.1$, and the beam profile in the far-field was nearly circular, see the inset in 349 Fig. 11(a).

The output performance and laser spectra of five $Er^{3+}:MF_2$ crystals are directly compared in 350 Fig. 11(c,d) using the same output coupling ($T_{OC} = 1.7\%$). The slope efficiency gradually 351 decreased in the sequence $Er^{3+}:CaF_2 \rightarrow Er^{3+}:SrF_2$ and Sr-containing crystals $\rightarrow Er^{3+}:BaF_2$, while 352 the laser threshold was in the range of 17 - 28 mW for all the crystals, being only slightly higher 353 354 for Ba-containing ones. The laser emission occurred at 2792 - 2800 nm, except of Er³⁺:SrF₂ for 355 which the laser operated at shorter wavelengths, 2730 and 2747 nm. The output characteristics 356 of mid-infrared Er³⁺:MF₂ lasers are summarized in Table 6. More details about the 2.8 µm laser 357 performance of Ba-containing crystals can be found in [44].



Table 6. Output Characteristics^a of Mid-Infrared $Er^{3+}:MF_2$ Lasers ($T_{oc} = 1.7\%$)

Parameter / Crystal	CaF_2	(Ca,Sr)F ₂	SrF_2	(Sr,Ba)F ₂	BaF_2
t, mm	6.81	6.99	6.53	6.99	6.66
$\lambda_{\rm P}$, nm	967.8	969.7	969.5	971.4	971.2
$\eta_{\rm abs}, \%$	81.9	84.3	76.1	83.3	83.8
P _{out} , mW	596	466	443	519	350
$\lambda_{\rm L}$, nm	2800	2796	2747	2794	2792
$P_{\rm th},{\rm mW}$	20	17	20	28	26
$\eta, \%$	30.4	24.8	23.5	25.0	17.2

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 ${}^{a}t$ – crystal thickness, $\lambda_{\rm P}$ - pump wavelength, $\eta_{\rm abs}$ – pump absorption under lasing conditions, $P_{\rm out}$ - output power, $\lambda_{\rm L}$ – laser wavelength, $P_{\rm th}$ – laser threshold, η – slope efficiency.

361 6. Conclusions

362 Fluorite-type Er³⁺:MF₂ parent and solid-solution crystals feature low-phonon-energy behavior, 363 very broad absorption and mid-infrared emission spectral bands, owing to the profound Er^{3+} ion 364 clustering and long ${}^{4}I_{11/2}$ and ${}^{4}I_{13/2}$ luminescence lifetimes. As for the "mixed" compounds, their 365 advantage is the lower melting points with respect to the corresponding parents. Considering the high thermal conductivity of these materials, the Er³⁺:MF₂ crystals are very promising for the 366 367 development of power-scalable and broadly tunable low-threshold mid-infrared lasers emitting at ~2.8 μ m. Based on a detailed comparative spectroscopic study of five 5 at.% Er³⁺:MF₂ fluorite-368 type crystals, including the parent compounds CaF₂, SrF₂, BaF₂, and "mixed" ones, (Ca,Sr)F₂ 369 370 and (Sr,Ba)F₂, the following conclusions are derived:

(i) The phonon energy of $Er^{3+}:MF_2$ crystals monotonously decreases with the square root of the M²⁺ cationic mass, from 321 cm⁻¹ ($Er^{3+}:CaF_2$) to 242 cm⁻¹ ($Er^{3+}:BaF_2$). Such a low-phonon energy behavior is a prerequisite for almost vanishing multiphonon non-radiative path from both the ⁴I_{11/2} and ⁴I_{13/2} Er^{3+} manifolds, as confirmed by the luminescence decay studies and the Judd-Ofelt analysis yielding the radiative lifetimes;

(ii) In the $M = Ca \rightarrow Sr \rightarrow Ba$ series, the absorption and mid-infrared emission spectra gradually become narrower and more structured, which is linked to the decreasing complexity of Er^{3+} clusters, and the luminescence lifetimes of the ${}^{4}I_{13/2} / {}^{4}I_{11/2} Er^{3+}$ manifolds increase, from 8.06 / 8.85 ms ($Er^{3+}:CaF_2$) to 9.17 / 10.36 ms ($Er^{3+}:BaF_2$) because of a decrease in the crystal 380 field strength. The observed ratio of the upper-to-lower laser level lifetimes and their values are 381 favorable for low-threshold mid-infrared laser operation;

382 (iii) The Er^{3+} ions in "mixed" crystals tend to reside in a local environment predominantly composed of the larger / heavier M^{2+} cations, leading to a great similarity between the spectra 383 384 of Er^{3+} : SrF₂ and Er^{3+} : (Ca, Sr)F₂, Er^{3+} : BaF₂ and Er^{3+} : (Sr, Ba)F₂. At LT, the spectra of Er^{3+} ions 385 in solid-solution crystals exhibit a notable inhomogeneous broadening;

(iv) For the doping level of 5 at.% Er^{3+} in MF₂ crystals, the LT spectroscopy reveals the 386 387 existence of a single class of Er^{3+} clusters with rather close absorption / emission properties (D 388 centers), contrary to crystals with low doping levels subject to ion clustering of various nature 389 (D(1) and D(2)).

390 In the present work, we employed high-brightness pumping to reveal the potential of 391 Er^{3+} :MF₂ crystals for efficient lasing at ~2.8 μ m. Further power scaling is envisioned by using 392 powerful InGaAs laser diodes as pump sources which is feasible owing to the good thermal 393 properties of these compounds. Further improvement of the slope efficiency, especially for Sr 394 and Ba-containing crystals should involve an optimization of the Er^{3+} doping level for boosting 395 the ETU efficiency. One hypothesis here is that a reduction in the cluster complexity may lead to weaker energy-transfer processes. Another idea is the Er³⁺, Pr³⁺ codoping for quenching the 396 397 metastable Er^{3+} lower-laser level (⁴I_{13/2}).

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- 401 **Data availability.** Data underlying the results presented in this paper are not publicly available at this time but may 402 be obtained from the authors upon reasonable request.

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