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# CONCENTRATION QUENCHING OF THE EMISSION OF $F_3^+$ AND $F_2$ COLOR CENTERS IN LiF

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## CONCENTRATION QUENCHING OF THE EMISSION OF $F_3^+$ AND $F_2$ COLOR CENTERS IN LiF

### Riassunto

Abbiamo studiato l'efficienza quantica di emissione dei centri  $F_3^+$  e  $F_2$  in funzione della loro concentrazione in cristalli di LiF colorati con elettroni da 3 MeV. Dall'analisi degli assorbimenti e delle luminescenze di tali centri, da 80 K a RT, abbiamo ricavato che la loro efficienza  $h$  è costante fino a concentrazioni di  $\sim 10^{16}$  centri/cm<sup>3</sup>, mentre oltre i  $10^{16}$  centri/cm<sup>3</sup>  $h$  decresce, leggermente per i centri  $F_2$  e sensibilmente per gli  $F_3^+$ . Inoltre, l'efficienza di questi ultimi difetti decresce ulteriormente all'aumentare della temperatura. Si suppone l'esistenza di un processo attivato termicamente, mentre i centri  $F_2$  ed altri difetti aggregati svolgono un ruolo importante.

### Abstract

*The emission efficiency of  $F_3^+$  and  $F_2$  centers as a function of their concentration has been investigated in LiF crystals colored at RT with 3 MeV electrons. From a careful investigation of their absorption and emission spectra from 80 K to RT, we discovered that the emission efficiency  $h$  of both  $F_3^+$  and  $F_2$  defects is constant up to  $\sim 10^{16}$  centers/cm<sup>3</sup>. When increasing the concentration up to  $10^{18}$  centers/cm<sup>3</sup>,  $h$  is decreasing slightly for the  $F_2$  centers and considerably for the  $F_3^+$  ones. Moreover, the efficiency of the latter centers further decreases by increasing the temperature. A thermally activated process is taking place, while the  $F_2$  and other aggregate defects play an important role.*

Key words: Color Centers; Lithium Fluoride; Emission Efficiency; Photoluminescence

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# CONCENTRATION QUENCHING OF THE EMISSION OF $F_3^+$ AND $F_2^-$ COLOR CENTERS IN LiF

## 1. INTRODUCTION

Among other alkali halides, Lithium Fluoride (LiF) occupies a special place because of its peculiar physical and optical properties. Unlike the other salts, it is moisture resistant, relatively hard and possesses a wide optical gap,  $\sim 12$  eV, from 0.1 to 7.0  $\mu\text{m}$ , which is an important prerequisite to investigate point defects and impurities. Moreover, it cannot be colored by the traditional additive method, but only by ionizing radiations, as x-rays,  $\gamma$ -rays, elementary particles and ions, which produce several absorption bands from the ultraviolet to the near-infrared region of the spectrum, due to various kinds of color centers (CC) [1]. Figure 1 shows the absorption spectrum of a LiF crystal colored with high energy electrons which, besides an intense F band, shows the presence of several bands referring to other color centers.

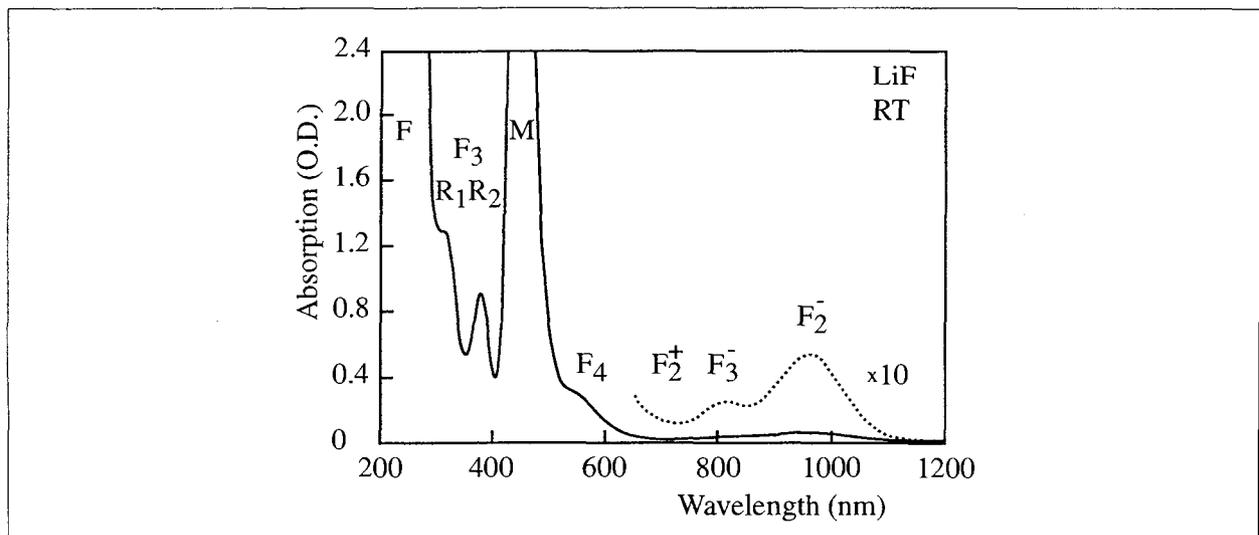


Fig. 1 - Absorption spectrum at RT of a LiF crystal, 0.5 mm thick, colored at RT with a dose of  $1.5 \times 10^{22}$  eV/cm<sup>3</sup> of 3 MeV electrons

It is worthwhile to note that, although the investigations started long ago [2], up to now no photoluminescence originating unambiguously from the F centers has been detected [3]. However, as a compensation for this scientifically painful failure, there is a rich zoology of aggregate CCs as  $F_2$ ,  $F_2^+$ ,  $F_2^-$ ,  $F_3^+$ ,  $F_3^-$  etc., all of which produce efficient emissions which have been extensively used for both passive and active optical devices [4, 5]. Figure 2 shows schematically the absorption and the emission of the centers which are known to emit up to now. In Table 1 all the known color centers in LiF are reported with the parameters of the absorption and emission bands at RT.

In Fig. 1 the F and M bands are strong enough to saturate the spectrum, and so we show the absorption of a less concentrated sample in Fig. 3a. Upon optical excitation at  $\sim 450$  nm the M absorption curve displays two distinct emission bands centered at  $\sim 540$  nm and  $\sim 680$  nm, which correspond to  $F_3^+$  and  $F_2$  centers, respectively, see Fig. 3b. The efficiencies of these two emissions are very high, although they suffer from some undesirable effects. The  $F_2$  emission fades away under intense optical pumping, if appropriate countermeasures are not provided. The  $F_3^+$  emission decreases under optical pumping, but after undergoing different time evolutions which depend on the temperature of the sample and the intensity of the pumping source, as shown in Figs. 4a and b, recovers fully its original value under dark conditions. Such reversible optical behavior has been studied in details recently [13] and a

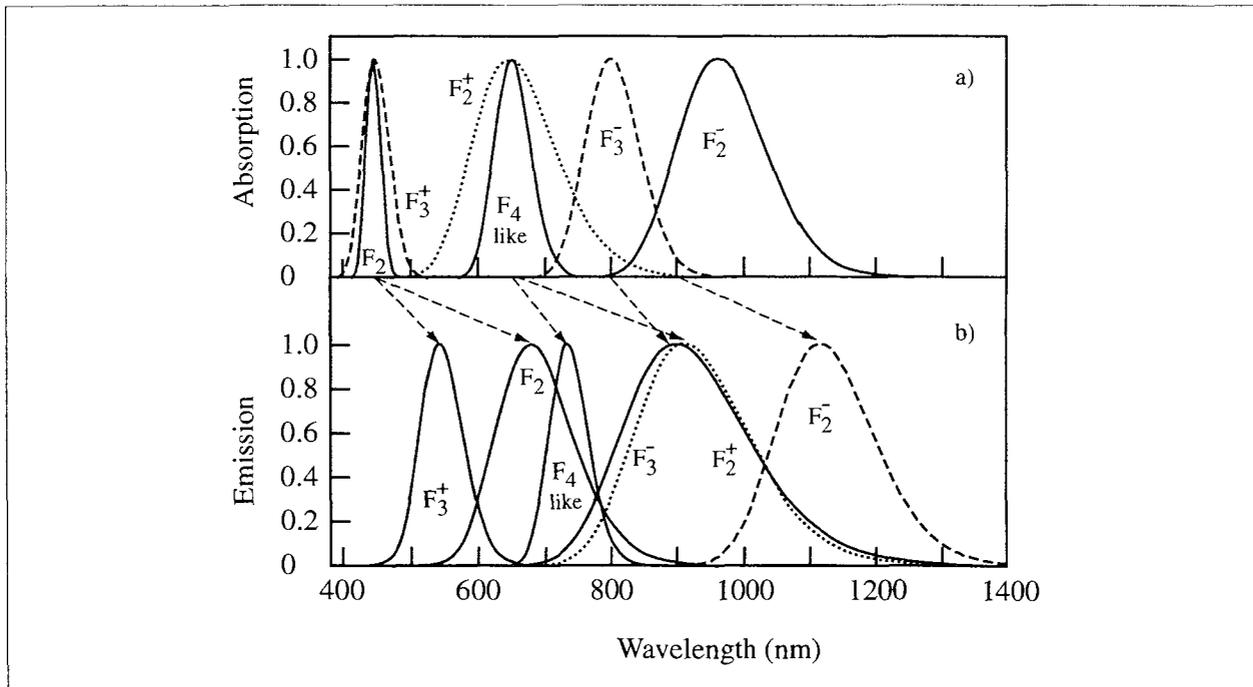


Fig. 2 – Absorption (a) and emission (b) bands, outlined as normalized gaussian curves, of the known color centers which possess photoluminescence in LiF crystals

**Table 1 - Color centers in LiF and their optical parameters at RT**

Center	$E_a$ (eV)	$\lambda_a$ (nm)	$E_e$ (eV)	$\lambda_e$ (nm)	$HW_a$ (eV)	$HW_e$ (eV)	$\tau$ (ns)
F	5.00[a]	248			0.76[a]		
$F_2$	2.79[b]	444	1.83[b]	678	0.16[b]	0.36[b]	15.5[c]
$F_3^+$	2.77[b]	448	2.29[b]	541	0.29[b]	0.31[b]	8.1[c]
$F_2^+$	1.92[d]	645	1.36[d]	910	0.43[d]	0.29[d]	15[d]
$F_2^-$	1.29[d]	960	1.11[d]	1120	0.21[d]	0.17[d]	65-105[d]
$F_3(R_1)$	1.92[e]	316					
$F_3(R_2)$	3.31[e]	374					
$F_3^-(R_1^-)$	1.88[e]	660					
$F_3^-(R_2^-)$	1.55[e]	800	1.38[e]	900	0.19[e]	0.33[e]	
$F_4(N_1)$	2.4[f]	517					
$F_4(N_2)$	2.26[f]	547					
$F_4$ -like	1.91[g]	650	1.69[g]	735	0.19[g]	0.16[g]	
Colloidi	2.75[e, f]	450					

[a] Ref. 6; [b] Ref. 7; [c] Ref. 8; [d] Ref. 9; [e] Ref. 10; [f] Ref. 11; [g] Ref. 12.

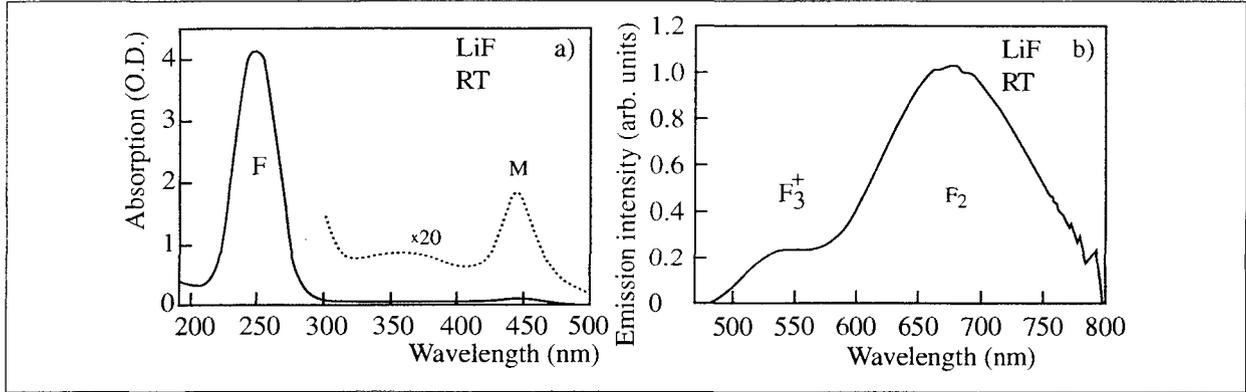


Fig. 3 - Absorption (a) and emission (b) spectra at RT of the LiF E6 sample, 1.85 mm thick, colored at RT with a dose of  $1.2 \times 10^{20}$  eV/cm<sup>3</sup> with 3 MeV electrons. The emission has been excited by the 458 nm line of an Ar laser

new state, the triplet state  $^3E_1$ , has been added to the usual four level scheme of the CCs, as shown in Fig. 5. It has been verified that  $1/\tau_0 > W_1 > W_2$ , where  $\tau_0$  is the radiative lifetime of the emitting state, and  $W_1$  and  $W_2$  are the probabilities for phonon-assisted radiationless transitions towards and from the triplet state; so the triplet state can be appreciably populated even under a continuous pumping rate  $U_0$  of low intensity, i.e.  $U_0 \ll 1/\tau_0$ . In this way a sizeable

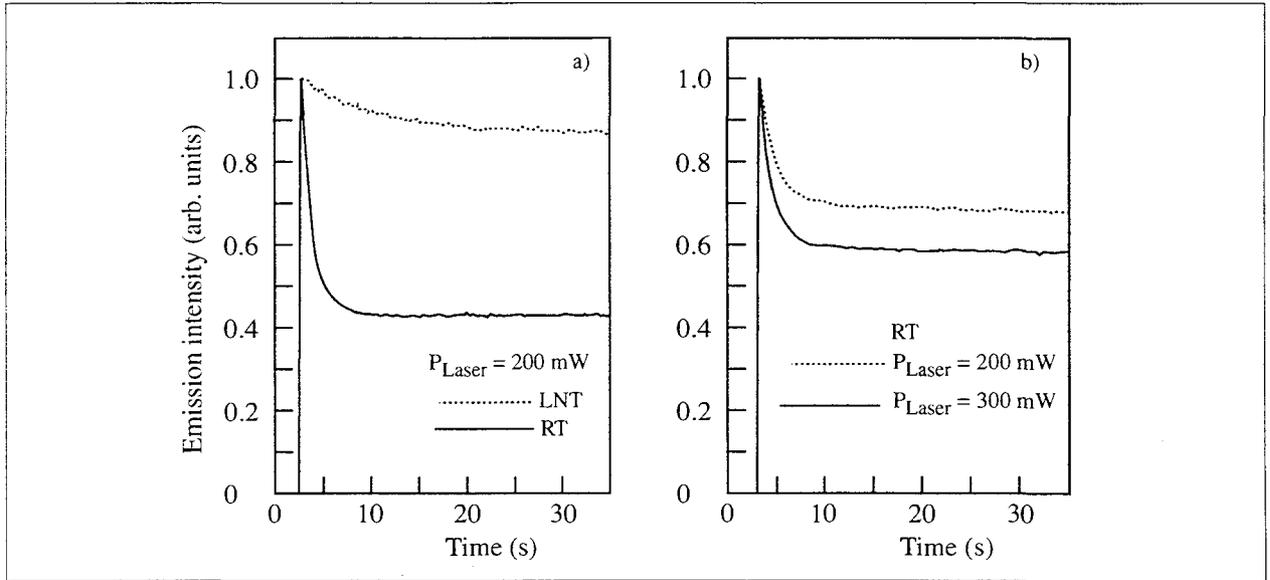


Fig. 4 - Time evolution of the emission at  $\lambda_{em}=520$  nm of a LiF crystal colored by 3 MeV electrons excited by the 458 nm line of an Ar laser, at different temperatures (a) and pumping powers (b). The results in (a) and (b) are not comparable because obtained in different experimental conditions

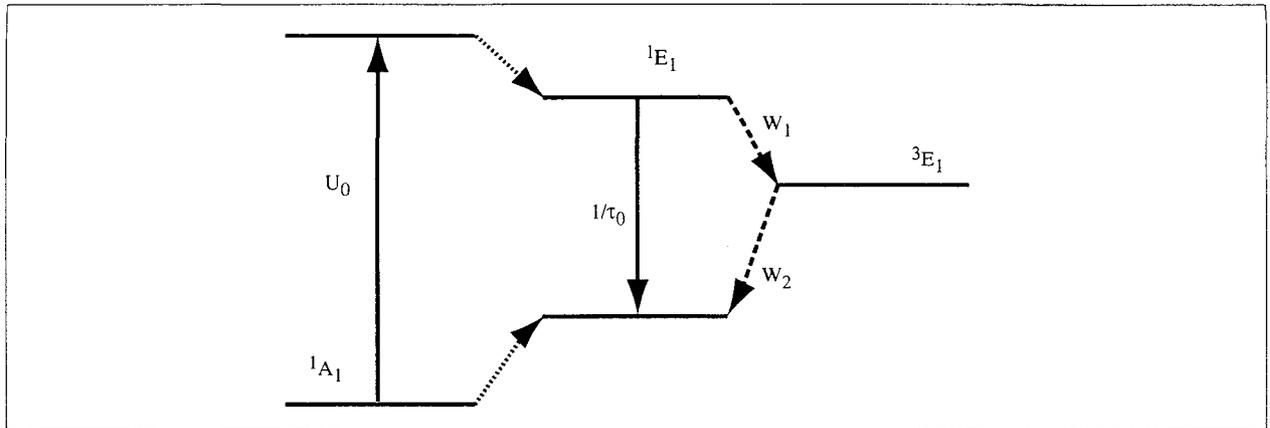


Fig. 5 - Optical cycle of the  $F_3^+$  centers

fraction of the  $F_3^+$  centers is subtracted to the basic optical cycle and the emission decreases, as in Figs. 4a and b. However, the oscillator strength of the transition  ${}^1E_1 \rightarrow {}^1A_1^*$ , where the star refers to the unrelaxed state, is so big that a pulsed laser can be operated also at RT [14].

Lately, the use of appropriately colored LiF as photonic material for integrated optics has been taken in serious consideration [15, 16]. In particular, narrow stripes of LiF, in the form of bulk and film, and colored with low energy electrons for a depth of  $\sim 1$   $\mu\text{m}$ , have been investigated for light guiding and amplification with success [17, 18]. However, in such cases



it is necessary to increase the concentration of the  $F_2$  and  $F_3^+$  centers well beyond the amount employed for the usual experiments in crystals, as those reported in Fig. 3, which is about  $10^{16}$  CCs/cm<sup>3</sup>. Preliminary results in optically thick systems have shown that the dynamical effects as those in Fig. 4 cannot be explained by the simple optical cycle of Fig. 5 and by the nonlinear optical process deriving from the high concentration of CCs, but rather by new interactions between the centers themselves [19].

In this paper we have addressed the fundamental question of the role of the concentration of the CCs in determining the emission efficiency of  $F_3^+$  and  $F_2$  centers.

## 2. EXPERIMENTAL PROCEDURE

The samples have been cut in thickness from 0.5 to 2.0 mm from a boule of a single LiF crystal containing less than 1 ppm of impurities. They have been successively colored at RT with the electron beam of the 3 MeV LINAC accelerator of the ENEA Laboratories of Frascati. The dose absorbed has been varied from  $10^{19}$  eV/cm<sup>3</sup> for the less colored samples to  $10^{23}$  eV/cm<sup>3</sup> for the heavy colored ones. The colored crystals were kept in the dark, to avoid possible photostimulated effects, up to the moment they were mounted in a cryostat.

Figures 6a and b show the absorption spectra at RT and LNT of a heavily colored sample,

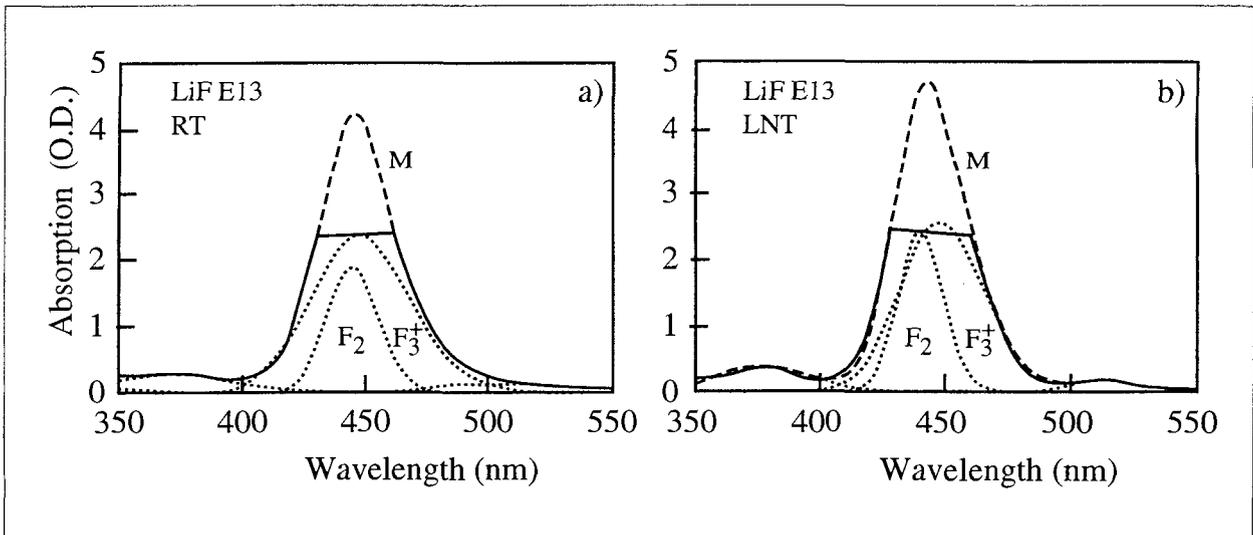


Fig. 6 - Absorption spectrum at RT (a) and LNT (b) of the LiF E13 sample, 1.4 mm thick, colored at RT with a dose of  $2.5 \times 10^{21}$  eV/cm<sup>3</sup> of 3 MeV electrons. The dashed and dotted curves represent a best fit simulation of the M,  $F_3^+$  and  $F_2$  absorption bands

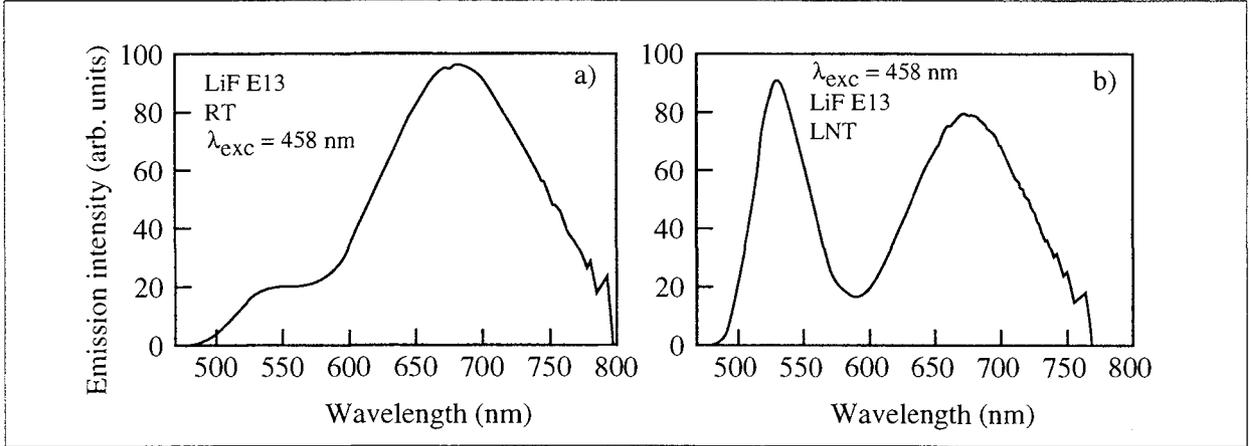


Fig. 7 - Emission spectrum at RT (a) and LNT (b) of the LiF E13 sample, 1.4 mm thick, colored at RT with a dose of  $2.5 \times 10^{21}$  eV/cm<sup>3</sup> of 3 MeV electrons. The emission has been excited by the 458 nm line of an Argon laser

where the M band is saturated. Under quasi-continuous optical excitation with the 458 nm line of an Ar laser, the luminescence has been spectrally analyzed in a collinear geometry and the emission spectra at RT and LNT have been reported in Figs. 7a and b.

The determination of the emission efficiency from such measurements has proved to be a task much more complex than thought at first. Indeed, especially in heavily colored samples like the one shown in Fig. 6, often the strong M absorption cannot be measured experimentally because of instrumental saturation effects. So, we had to reconstruct the missing part of the absorption spectrum by resorting to our knowledge of the band parameters [7] and fitting procedures with well tested computer programs. The dotted lines in Fig. 6 show the final result of this kind of calculations. In the present case, also the overlapping of the  $F_3^+$  and  $F_2$  bands had to be resolved in order to correctly estimate the concentration of the  $F_3^+$  and  $F_2$  centers. Indeed, from the maximum value of the absorption coefficient  $\alpha_{\max}$ , the concentration has been calculated via the known Smakula formula for gaussian-shaped bands:

$$N(\text{cm}^{-3}) = 0.87 \times 10^{17} \frac{n}{(n+2)^2} \frac{W(\text{eV})}{f} \alpha_{\max}(\text{cm}^{-1}), \quad (1)$$

where  $n=1.396$  is the refractive index of LiF at  $\sim 445$  nm (the absorption peak position for both  $F_3^+$  and  $F_2$  defects),  $W$  is the full width at half maximum of the absorption band, and  $f$  is the oscillator strength. In Table 2 we have reported the values of the previous parameters as known from the literature.

Table 2 - Peak position and width of the absorption and emission bands, at RT and LNT, and oscillator strength, independent of temperature, for the F,  $F_3^+$  and  $F_2$  centers, as known from the literature and used in this work

Color center	F		$F_3^+$		$F_2$	
T (K)	296	77	296	77	296	77
Temperature						
$E_a$ (eV)	5.00[a]	5.14[b]	2.77[c]	2.77[c]	2.79[c]	2.82[c]
Absorption peak	5.00[b]					
$W_a$ (eV)	0.76[a]		0.25[c]	0.20[c]	0.13[c]	0.13[c]
FWHM	0.74[b]	0.70[b]				
$E_e$ (eV)			2.29[c]	2.33[c]	1.83[c]	1.84[c]
Emission peak						
$W_e$ (eV)			0.27[c]	0.18[c]	0.31[c]	0.25[c]
FWHM						
f	0.56[d]		0.5*		0.28[d]	
Oscillator strength						

[a] Ref. 6; [b] from our data; [c] Ref. 7; [d] Ref. 20.

\*The parameter is not known, so its value has been arbitrarily set at 0.5.

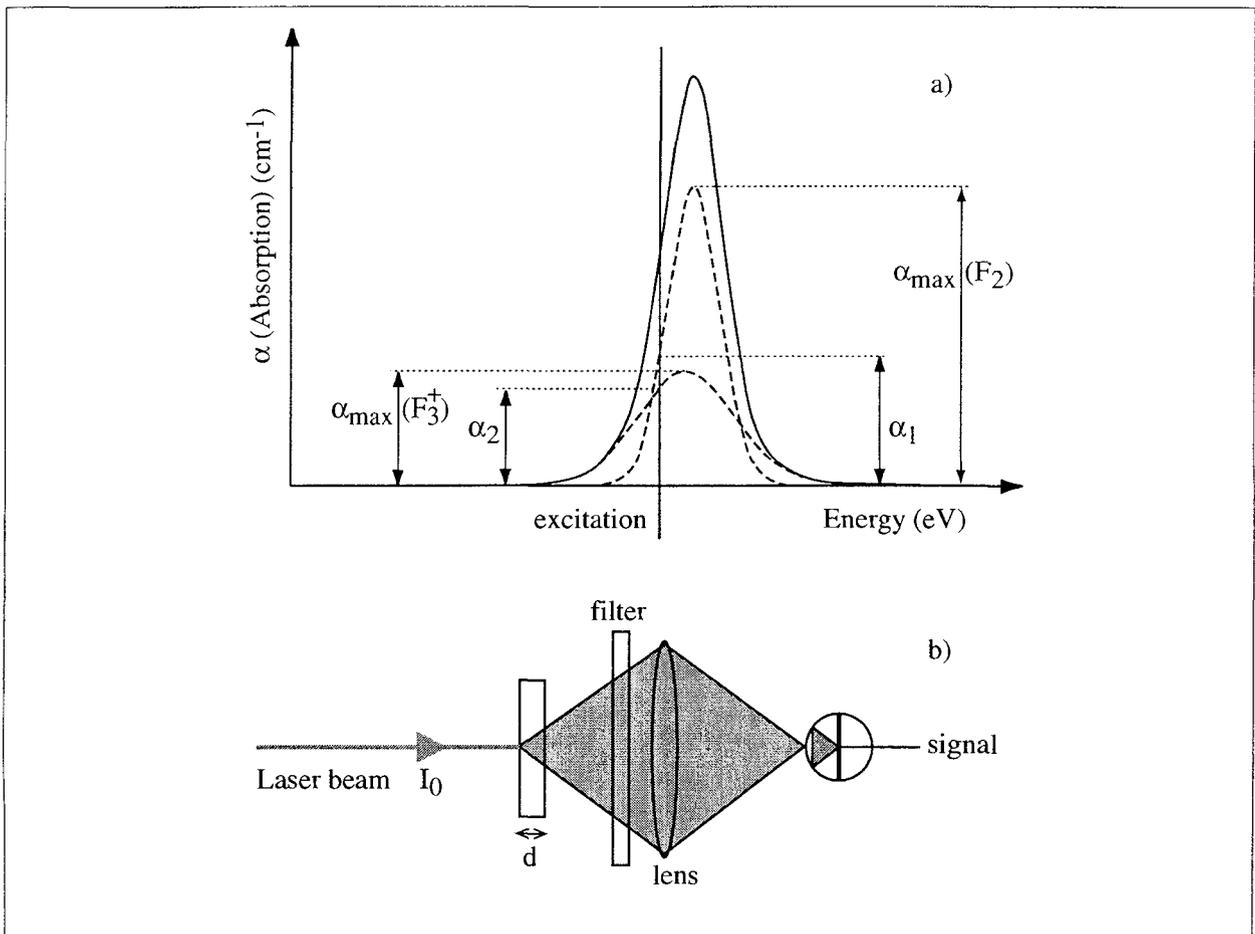


Fig. 8 - Schematic description of the parameters used in formula (2) (a) and a sketch of the optical experiment (b)

Anyway, to make a long story short, the final value of the efficiency  $\eta$  has been calculated with the following formula:

$$\eta = A \frac{L}{I_0 T} \frac{\alpha_1 + \alpha_2}{\alpha_{\max}} \frac{1}{\left(1 - e^{-(\alpha_1 - \alpha_2)/d}\right)}, \quad (2)$$

where  $A$  is an arbitrary coefficient,  $L$  is the peak intensity of the luminescence,  $I_0$  the intensity of the excitation beam on the sample and  $T$  is the transmission of the surfaces of the sample, which takes into account the reflection and the scattering if any. The absorption coefficients  $\alpha$  refer: to the  $F_2$  centers at the pumping wavelength  $\lambda_{\text{exc}}$ ,  $\alpha_1$ , to the  $F_3^+$  centers at  $\lambda_{\text{exc}}$ ,  $\alpha_2$ , and to the maximum absorption of the center under consideration,  $\alpha_{\max}$ , as illustrated in Figure 8, where also a sketch of the photoluminescence geometry experiment is shown. The coefficient  $A$  has been fixed arbitrarily in order that  $\eta=0.1$  on average at relatively low concentrations of color centers. No attempts have been made to measure absolutely the emission efficiency, although it is often reported to approach unity.

### 3. RESULTS AND DISCUSSION

Figure 9 shows the values of the relative emission efficiency,  $\eta$ , for the  $F_3^+$  centers as a function of their concentration at RT (a) and LNT (b). Figure 10 shows the same for the  $F_2$  centers.

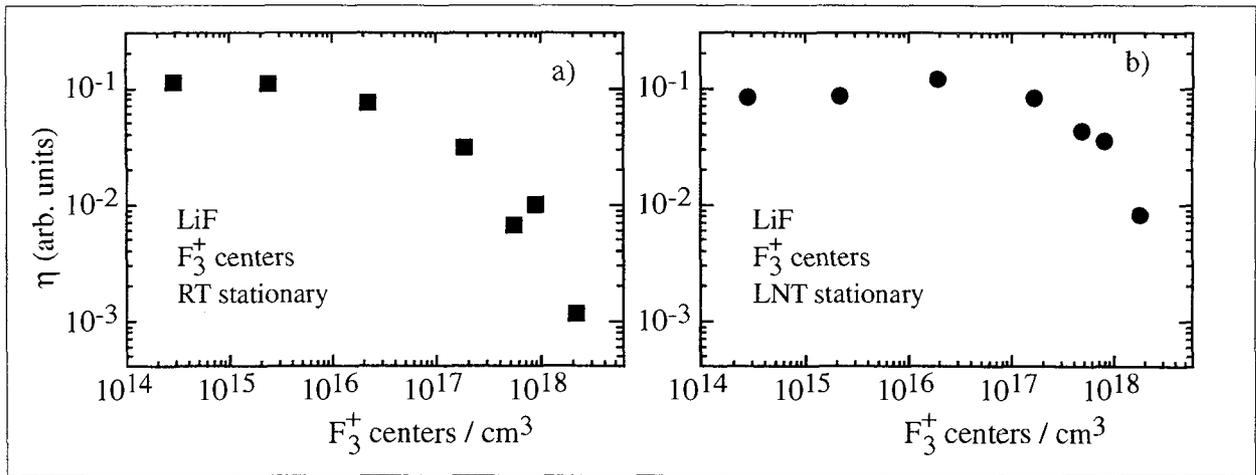


Fig. 9 - Relative quantum efficiency,  $\eta$ , of the  $F_3^+$  center emission as a function of the same  $F_3^+$  center concentration. The stationary luminescence is obtained by pumping with the 458 nm line of an Ar laser at RT (a) and LNT (b)

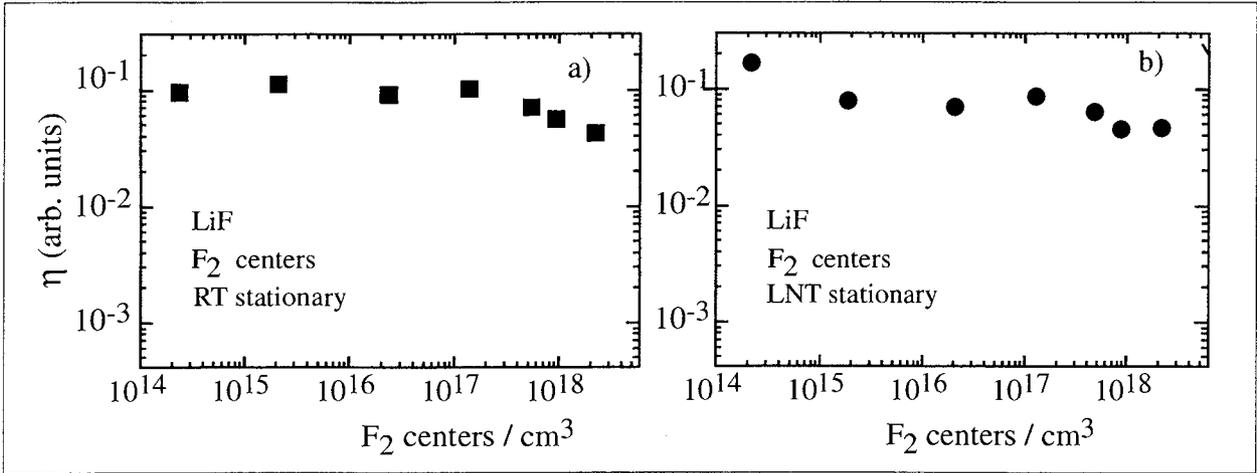


Fig. 10 – Relative quantum efficiency,  $\eta$ , of the  $F_2$  center emission as a function of the same  $F_2$  center concentration. The stationary luminescence is obtained by pumping with the 458 nm line of an Ar laser at RT (a) and LNT (b)

At a first analysis, the emission efficiency is constant below  $\sim 10^{16}$  centers/ $\text{cm}^3$  for both  $F_3^+$  and  $F_2$  centers while, well above this value, it is decreasing slightly for the  $F_2$  centers and considerably for the  $F_3^+$  centers, much more at RT than at LNT.

Letting aside the absolute value of the phenomenon, on which we will come back later on, we asked ourselves why the concentration quenching of the  $F_3^+$  centers should be so different at RT with respect to LNT. Indeed, it is well known that several important parameters of the  $F_3^+$  centers do not depend on the temperature, notably the lifetime [8], or depend on it very slightly, as the FWHM of the absorption and emission bands [7], see Table 2. The only effect at our knowledge strongly dependent on the temperature is the dynamic behavior of the  $F_3^+$  center population [13, 16], already shown in Fig. 4. So, is the triplet state able to influence besides the population also the intensity of luminescence by still unknown mechanisms? In order to check this hypothesis we performed new experiments on the same samples utilized for the measurements of Fig. 9. Indeed, the values reported there have been taken in a steady state of excitation, i.e. after the dynamical effect of Fig. 4 has reached the equilibrium value for  $t \rightarrow \infty$ . But at that time the triplet state is fully populated and this sizeable population could interact with the one still participating to the active optical cycle. However, this is not the case at the beginning,  $t=0$ , when the triplet state is completely empty. So, this time in formula (2) we used the initial value of the  $F_3^+$  luminescence and, however, also the initial absorption, in order to get the real value of the emission efficiency. The results, reported in Fig. 11, are very similar within the experimental errors to those reported in Fig. 9, and so they exclude the

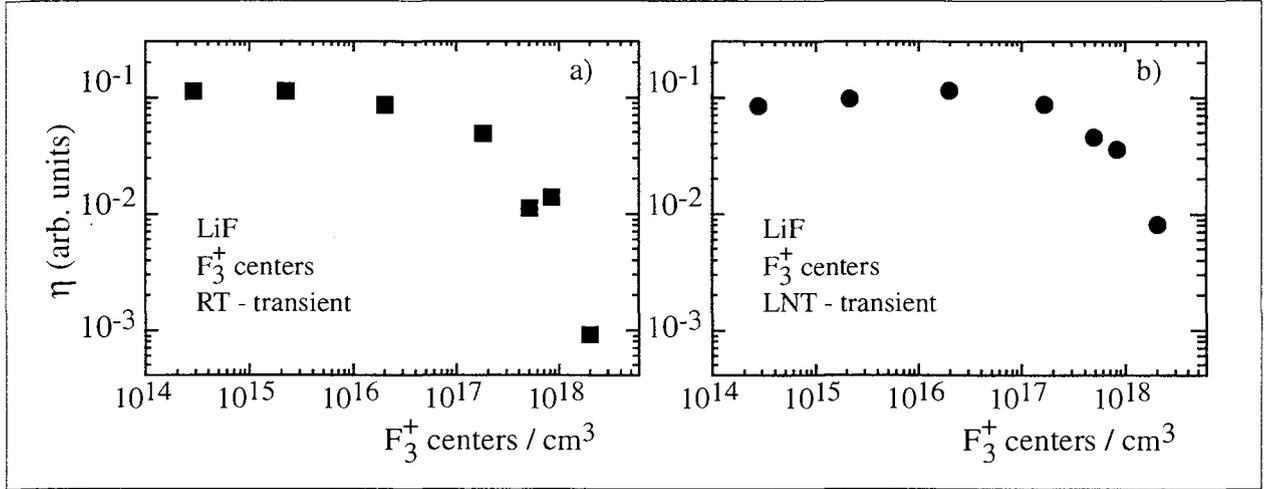


Fig. 11 - Quantum efficiency,  $\eta$ , of the  $F_3^+$  center emission as a function of the same  $F_3^+$  center concentration. The initial luminescence is obtained by pumping with the 458 nm line of an Ar laser at RT (a) and LNT (b)

hypothesis that the triplet state is of some influence for the observed  $F_3^+$  concentration quenching phenomenon.

Now, let us go back to the matter of the relative values of the efficiency, and especially the reliability of the measurements at high concentrations. Indeed, we realized during the measurements that some other parameters should be added to the formula (2) in order to take into account different kinds of absorption. Fig. 12 shows the details of the absorption of the most colored sample considered in this work, corresponding to the last dot at the right of Figs. 9, 10 and 11. The shoulder above 500 nm corresponds to the  $N_1$  and  $N_2$  bands, which are usually absent in less concentrated samples. On the same spectrum we have simulated with dotted lines the profiles of the  $F_3^+$  and  $F_2$  emission bands. It is evident that while the reabsorption effect on the  $F_2$  luminescence is negligible, this is not the case for the  $F_3^+$  luminescence. The final effect of this further correction is not easy to be quantified exactly, but we can calculate approximately the attenuation of the  $F_3^+$  luminescence at its peak value of 541 nm. Without going into details, this preliminary correction moves the last points of Figs. 9a and 9b to  $\sim 10^{-2}$  and  $\sim 10^{-1}$ , respectively. At moment, we did not apply to the other samples the same kind of correction, which however should be smaller for lower concentrations of defects, but it does not eliminate completely the sizeable quenching of luminescence of the  $F_3^+$  centers at RT.

At this point, it is also worthwhile to remind that the emission efficiency of the  $F_3^+$  centers is starting to decrease more or less at the same concentration where we have recently observed

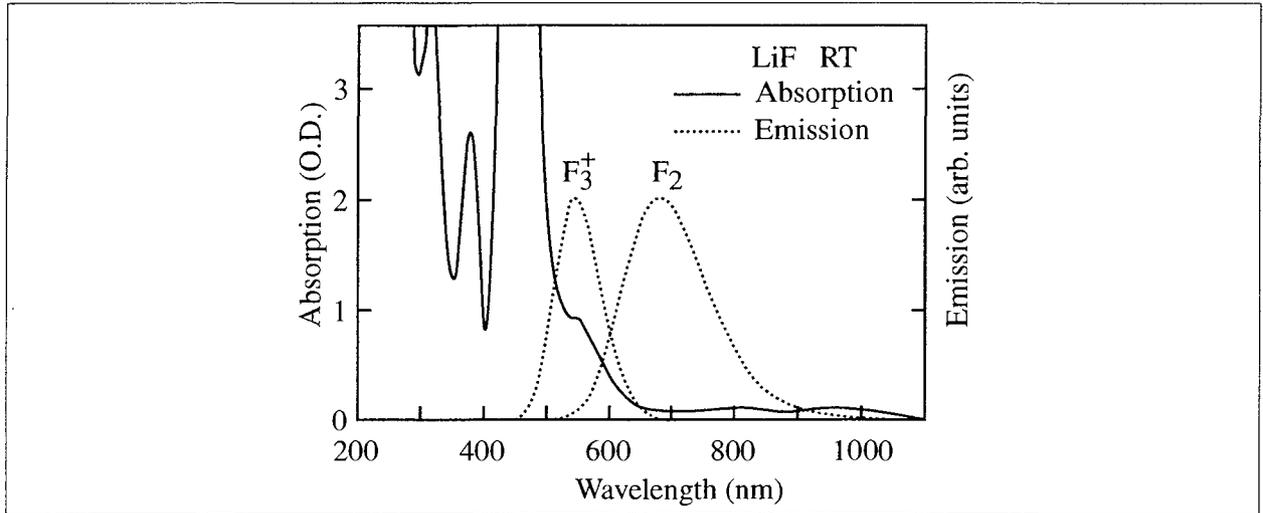


Fig. 12 - Absorption spectrum (full line) at RT of the most colored sample used in this work, 0.5 mm thick and irradiated with  $1.2 \times 10^{23}$  eV/cm<sup>3</sup> of 3 MeV electrons. The dotted lines represent schematically the emission bands of the  $F_3^+$  and  $F_2$  centers

anomalous dynamic behaviors of the emission [19], which were tentatively ascribed to an interaction between the existing centers, i.e.  $F_3^+ \leftrightarrow F_3^+$  or  $F_3^+ \leftrightarrow F_2$ .

#### 4. CONCLUSIONS

In this work we have shown that there is a slight decrease of the emission efficiency for the  $F_2$  centers at concentrations around  $10^{18}$  CCs/cm<sup>3</sup>, independent of temperature. At the same time the  $F_3^+$  emission efficiency decreases at RT above  $10^{16}$  CCs/cm<sup>3</sup>, while at LNT the efficiency seems to be constant up to  $10^{17}$ - $10^{18}$  CCs/cm<sup>3</sup>. The triplet state, or better the  $F_3^+$  population trapped in it, does not have any effect on the quenching phenomenon.

On the other hand, the existence of a temperature dependence of  $\eta$  for  $F_3^+$  centers is the onset signature of a thermally activated process, and the fact that it is related to concentrations higher than  $10^{16}$  CCs/cm<sup>3</sup> tells us that an interaction between two centers is going on, and it depends on their distance. As much as we know up to now, we have to take into account possible interactions between the centers themselves,  $F_3^+ - F_3^+$ , and with other centers, for instance  $F_3^+ - F$ , the latter centers two orders of magnitude more numerous. However, we

cannot exclude interactions with other less numerous centers like  $F_3$ ,  $F_2$ ,  $F_4$ ,  $F_2^+$ ,  $F_3^-$  and  $F_2^-$ , which are always present in the samples, as shown in Fig. 1.

As far as the  $F_2$  centers are concerned, it is worthwhile to remind that they are well known killers of the F center emission in alkali halides [21]. Unfortunately, the samples used for the present work were all colored at RT and then the  $F_3^+/F_2$  population ratio is constant in all samples, so there is no possibility to find out if there is any omo- and/or etero-interaction. However, we know that it is possible to change the  $F_3^+/F_2$  ratio by coloring at different temperatures with beams of electrons [22]. On this respect we started to color several LiF crystals at 213 K, and preliminary measurements show that the results up to  $10^{16}$  centers/cm<sup>3</sup> coincide with those reported here, while above this concentration there is some discrepancy which discloses some effects of the  $F_2$  centers, although it is not yet statistically significant.

In conclusion, we have observed a slight decreasing of the emission efficiency for  $F_2$  centers up to  $10^{18}$  centers/cm<sup>3</sup>, independent from the temperature. At the same time the  $F_3^+$  centers display a much more sizeable effect, especially at RT. These experiments suggest for the  $F_3^+$  centers the onset of an interaction among centers which affects the fundamental parameters of the optical cycle. Further experiments should be performed to assess more firmly this hypothesis.

It is clear that there is much work to be done before we can fully understand the just described quenching phenomena. First of all, the experimental data of  $\eta$  should be corrected in the proper way in order to eliminate any effect of residual absorption on the emitted photoluminescence, which is under way. Secondly, the efficiency of luminescence should also be measured indirectly through the lifetime of the emission, which is a well known method in optical ions in insulating materials [23, 24, 25] and does not suffer from secondary effects, except self trapping which is usually absent in color centers.

Moreover, if energy exchanges occur among different centers, also new emissions due to the acceptor centers should be measured in case of radiative transitions. Finally, heavily colored samples containing different amounts of other aggregate centers should be prepared in order to single out the CCs responsible for the quenching.

If this ordeal should be accomplished, it would be possible to prepare colored crystals which are immune, as much as possible, to the quenching of luminescence, an unwanted phenomenon especially for low dimensionality structures which are considered nowadays for miniaturized optoelectronic devices.



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