

## RADIATIVE TRANSITIONS IN $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ NANOPOWDERS

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**Abstract-** This paper presents the results of experimental studies of the luminescence spectra of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ferrite nanopowders with different contents of Zn and Ni at a temperature of 300K. A xenon lamp and a YAG-Nd laser ( $\lambda = 532$  nm) were used to excite luminescence. It is shown that the obtained spectra can presumably be interpreted within the framework of models [1-7], models used to interpret optical and other spectra, for example, magnetite, a structural analogue of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ferrites.

**Keywords:** Photoluminescence, Ni-Zn Ferrites, Sublattice, Superexchange.

### 1. INTRODUCTION

The results of experimental studies of the luminescence spectra of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ferrite nanopowders with various concentrations of Ni and Zn are not only of scientific interest but also relevant because of the high practical importance of these ferrites, which is confirmed by numerous publications and technical applications of rapidly developing modern technologies: energetics, nanoelectronics, information - computer systems for data processing, including creating and development of new types of various functional elements operating in environmental conditions that differ significantly in their aggressiveness. For example, because of exposure to intense solar radiation, the characteristics of a frequency-dependent resistor designed to damp high-frequency overvoltage [8] in power communication networks change due to the intense luminescence of the ferrite filter.

Note that a characteristic peculiarities of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  is ferrites, which, according to modern concepts [9-11], contain two collinear magnetic sublattices associated with joint superexchange interaction. The observed improvement of research methods, based on new experimental research equipment, made it possible to establish the presence of a very weak magnetic phase in  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  [12]. However, the intensity of the peaks corresponding to this phase was so weak that it turned out to be impossible to separate them from the intense peaks of iron ions of the A and B sublattices, and, accordingly, the nature and parameters of the weak magnetic sublattice were not established.

The main purpose of this work was to study the photoluminescence spectra of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ , to study magnetic excitations, optical properties, and parameters of the weak magnetic phase in nanopowders of these materials. Note that this work is a continuation of the research, the results of which were published in works [8, 13, 14].

### 2. SAMPLE PREPARATION

The  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  nanopowders are synthesized by the thermal sintering method of high purity NiO, ZnO and  $\text{Fe}_2\text{O}_3$  compounds followed by annealing for 2 hours at 960°C [15]. The diameters of nanoparticles of all compositions are about 20-40 nm. The quality of nanopowders monitored by X-ray diffractograms and optical methods. Lattice distortions have little effect on Raman spectra. Detailed X-ray studies of the formation of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ferrite films have shown that the process of their formation, as pointed in [16], goes through three stages: at the first stage  $\text{ZnFe}_2\text{O}_4$  is obtained, while part of NiO and  $\text{Fe}_2\text{O}_3$  remain in the free state; in second stage the process of including  $\text{Ni}^{2+}$  ions in the  $\text{ZnFe}_2\text{O}_4$  lattice begins and compound with an excess of Ni content are formed against stoichiometry; in the third stage the composition compound is finally formed. All observed changes are in good agreement with changes in the content of  $\text{Fe}^{3+}$  cations [17] in the compositions of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  films. All observed processes are in good agreement with changes in the content of bivalent and trivalent cations of iron [17], nickel, and zinc in nanopowders.

We note that it established in work [18] that the most homogeneous composition of  $\text{ZnFe}_2\text{O}_4$ , accompanied by the largest incorporation of Fe ions into the ZnO structure, achieved when using  $\alpha\text{-Fe}_2\text{O}_3$  powders. A significantly smaller amount of Fe is included into the ZnO structure in samples obtained based on FeO and  $\text{Fe}_3\text{O}_4$ . The spatial symmetry group of  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  corresponded to Fd3m. However, all  $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$  ferrite compositions, except  $\text{ZnFe}_2\text{O}_4$ , referring to normal spinel ( $x = 1$ ), have a reverse spinel structure, a good idea of which for the  $\text{NiFe}_2\text{O}_4$  case ( $x = 0$ ) can be seen in Figure 1 [19-22].

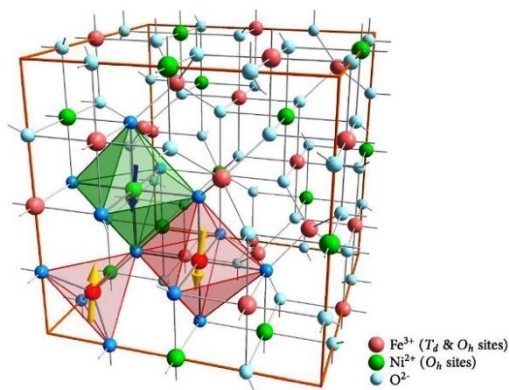


Figure 1. The unit cell of the inverse spinel lattice of NiFe<sub>2</sub>O<sub>4</sub>: Fe<sup>3+</sup>-cations (red) are distributed equally across tetragonal (T<sub>d</sub>) and octahedral (O<sub>h</sub>) lattice sites, while Ni<sup>2+</sup>-cations (green) occupy O<sub>h</sub> sites. An antiferromagnetic coupling between the T<sub>d</sub> and O<sub>h</sub> sites compensates the magnetic moments of the Fe<sup>3+</sup>-cations, why only the Ni<sup>2+</sup>-cations account for the net macroscopic magnetization of 2 μ<sub>B</sub>/f.u [23]

### 3. RESULTS AND DISCUSSION

#### 3.1. Experimental Details

The luminescence spectra of all synthesized Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanopowders compositions studied on LS-55 spectrometer with a Monk-Giddison monochromator at room temperature in the 300-700 nm wavelength range.

The results of these studies are summarized in Figures 2-3 and Tables 1-2. Table 2 shows a comparison of the results obtained, averaged over the corresponding peaks of the luminescence spectra at different excitation energies, with those previously published in work [14], as well as the interpretation of the observed luminescence peaks.

The positions of the peaks of the luminescence spectra for all compositions in Tables 1-2 are averaged. In Figure 2 the following designations are used: A, B, C, etc. - luminescence spectra when excited from an Xe source: 280 nm, 290 nm, 300 nm, 325 nm, 350 nm, 375 nm, 388 nm, 400 nm, 425 nm; a, b, etc. - compositions: x = 0; 0.25; 0.4; 0.5; 0.6; 0.75; 1, respectively. The numbers 1, 2, in Table 1 indicate the energies and wavelengths of electronic transitions averaged over the studied compositions. The upper rows of the table cell are the peak wavelengths in nm, the lower rows are in eV. The luminescence spectra of all synthesized compositions of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanopowders were also studied on a confocal Raman spectrometer. These studies revealed the presence of a photoluminescence line in the 600-1000 nm region with a maximum at ~822 nm, the intensity of which depended on the composition. The maximum intensity of the luminescence band at an exciting radiation power of 10 mW was not reached.

#### 3.2. Discussion

Photoluminescence spectroscopy is an important method for studying electronic, optical and other properties, it provides information on the band gap of metal oxide and other compounds, with a relative active position and states in it. The band structure of magnetite

nanoparticles proposed in [1] indicates that the peak at about ~840 nm (1.47 eV) can be attributed to electron traps in the tetrahedral region of the structure associated with oxygen evacuations [1]. Their mutual correlation is beyond doubt and is a consequence of exchange processes between Fe<sup>2+</sup> → O<sup>e-</sup> → Fe<sup>3+</sup> ions.

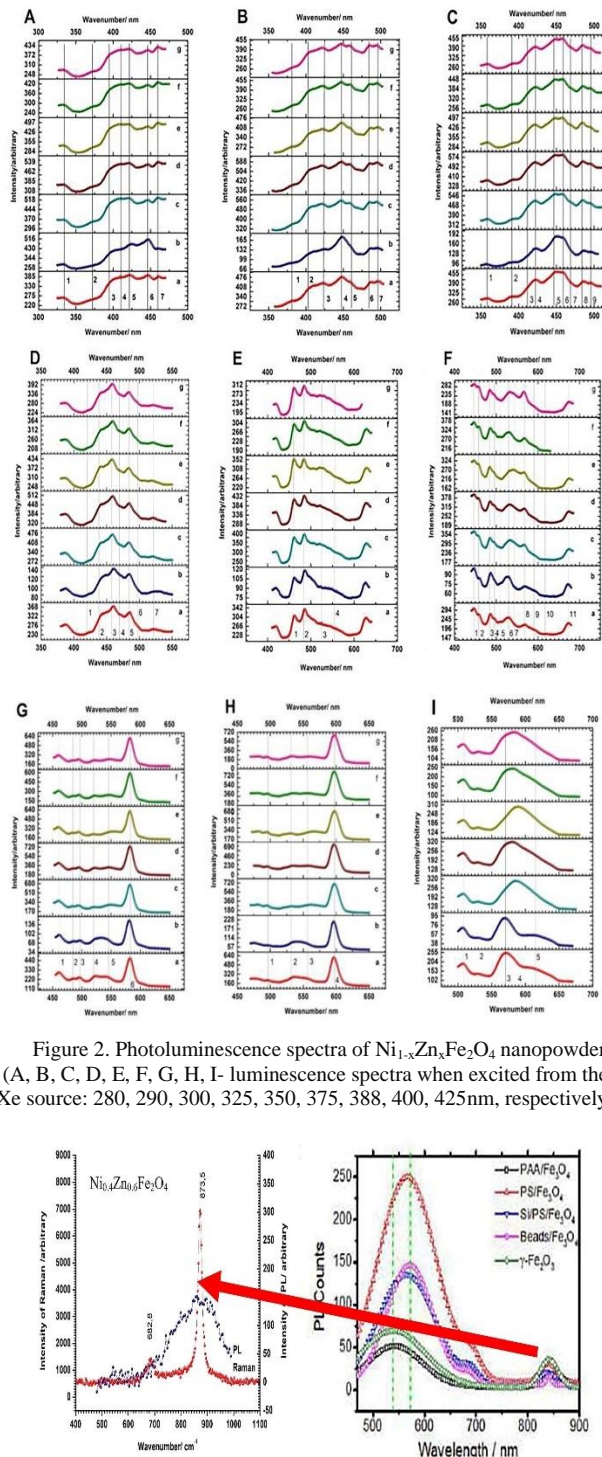


Figure 2. Photoluminescence spectra of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanopowders (A, B, C, D, E, F, G, H, I- luminescence spectra when excited from the Xe source: 280, 290, 300, 325, 350, 375, 388, 400, 425nm, respectively)

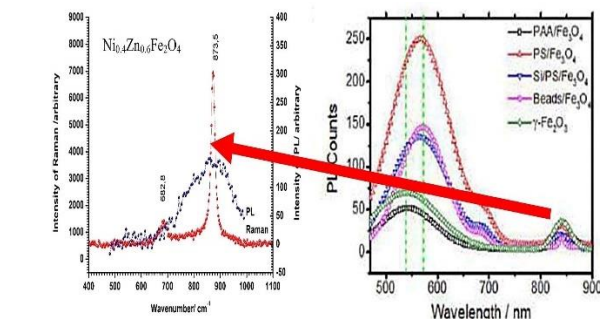


Figure 3. Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> thin film (a) [24] and Fe<sub>2</sub>O<sub>3</sub> (b) (840cm<sup>-1</sup>) [1] to the maximum of oxygen vacancies is indicated by a red arrow. The blue color indicates the components of the photoluminescence spectrum of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> nanopowders, and the red color indicates a maximum of 873 cm<sup>-1</sup> in the Raman spectrum of Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> thin films (a)

Table 1. Energies and wavelengths of electronic transitions averaged over the studied compositions

A	B	C	D	E	F	G	H	I
280nm 4.427eV	290nm 4.275eV	300nm 4.132eV	325nm 3.814eV	350nm 3.542eV	375nm 3.306eV	388nm 3.195eV	400nm 3.099eV	425nm 2.917eV
332 nm 3.734 eV	343 nm 3.614 eV							
372 nm 3.332 eV	367 nm 3.378 eV	358 nm 3.462 eV						
396 nm 3.13 eV	385 nm 3.22 eV	393 nm 3.154 eV	388 nm 3.195 eV					
406 nm 3.053 eV	410 nm 3.024 eV	410 nm 3.024 eV	410 nm 3.024 eV					
421 nm 2.945 eV	422 nm 2.938 eV	422 nm 2.938 eV	422 nm 2.938 eV	419 nm 2.959 eV				
446 nm 2.779 eV	448 nm 2.767 eV	448 nm 2.767 eV	448 nm 2.767 eV		446 nm 2.779 eV			
461 nm 2.689 eV	458 nm 2.707 eV	458 nm 2.707 eV	461 nm 2.689 eV	462 nm 2.683 eV	458 nm 2.707 eV	460 nm 2.685 eV		
	486 nm 2.551 eV	485 nm 2.556 eV	485 nm 2.556 eV	485 nm 2.556 eV	486 nm 2.551 eV	485 nm 2.556 eV	482 nm 2.572 eV	
	496 nm 2.499 eV	495 nm 2.504 eV	507 nm 2.445 eV	506 nm 2.45 eV	512 nm 2.421 eV	495 nm 2.504 eV	495 nm 2.504 eV	507 nm 2.445 eV
			522 nm 2.375 eV	521 nm 2.379 eV	527 nm 2.352 eV	522 nm 2.375	521 nm 2.379 eV	
			548 nm 2.262 eV	542 nm 2.287 eV	542 nm 2.287 eV	547 nm 2.266 eV	534 nm 2.321 eV	530 nm 2.339 eV
				558 nm 2.222 eV	567 nm 2.186 eV	564 nm 2.198 eV	565 nm 2.194 eV	
				598 nm 2.073 eV	592 nm 2.094 eV	583 nm 2.126 eV	597 nm 2.076 eV	589 nm 2.105 eV
				628 nm 1.974 eV	622 nm 1.993 eV			609 nm 2.036 eV
								636 nm 1.949 eV
					675 nm 1.837 eV			

It should be noted that the similarity of this maximum is also observed in the photoluminescence spectra obtained by the authors, for example, in [1, 25], as well as for Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> in this work. We used the band structure [1] to analyze the luminescence spectra.

In work [1], three main peaks were revealed: 540, 690 and 840 nm. According to [1, 22], the photoluminescence maximum at 550 nm (2.25 eV) [22] or 540 nm (2.3 eV) [1] is explained by the radiative recombination of electrons from T<sub>2g</sub> → E<sub>g</sub> (2.2 eV) in the octahedral site. A much weaker peak at ~ 690 nm (1.79 eV) corresponds to the recombination of trapped electrons from the octahedral site to the O (2p) tetrahedral site. As follows from works [22, 25], the intensity of the emission bands at 542 nm and 518 nm changes with increasing Zn/Ni substitution. Hence, it can be concluded that an increase in the Zn<sup>2+</sup> concentration corresponds to the situation when zinc ions occupying tetrahedral positions transfer Fe ions into octahedral positions, which, on the one hand, should lead to an increase in magnetization, on the other hand, to a decrease in the exchange interaction between the sublattices.

Note that despite the difficulty of the superexchange interaction process: Ni<sup>2+</sup> → O → Fe<sup>3+</sup>, there still exists and can make an additional contribution to the magnetic permeability. In the near infrared and visible regions of the luminescence spectra of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanopowders, as in γ-Fe<sub>2</sub>O<sub>3</sub> [6], two absorption bands are observed at

about 640 nm and 900 nm, which were interpreted by the authors of [6] as transitions: 6A<sub>1</sub> (6S) 4T<sub>2</sub> (4G) and 6A<sub>1</sub> (6S) 4T<sub>1</sub> (4G) ferric Fe<sup>3+</sup> iron, respectively. The same features were observed in the absorption spectra of goethite and lepidocrocite single crystals [6]. Thus, the group of peaks at 627 nm, 636 nm, and 673 nm is the expected splitting of Fe<sup>3+</sup> levels in the tetrahedral field of the crystal structure of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> ferrite. A similar group of peaks was also observed in the band at about 800 nm of the luminescence spectrum of Fe<sub>3</sub>O<sub>4</sub>. Hence, it should be taken into account that the peak at 673 nm, as well as at 840 nm, can be attributed to Fe<sup>2+</sup>, and in the case of 840 nm, in the octahedral site.

The peak at 665 nm in the luminescence spectrum of ZnFe<sub>2</sub>O<sub>4</sub> [26] (Table 2) apparently has a different nature from the model used above: <sup>4</sup>A<sub>2</sub>(F) → <sup>4</sup>T<sub>1</sub>(P); <sup>6</sup>A<sub>1g</sub> → <sup>4</sup>T<sub>1g</sub>(G); <sup>0</sup>Fe<sup>2+</sup> ↔ <sup>0</sup>Fe<sup>3+</sup>. Spectral bands are shorter than 600 nm are present in the diffuse reflectance spectra of all iron oxides. Of the two characteristic bands around 480-600 nm and 430 nm, the first at 550 nm is an indication of the contribution of hematite to the spectrum. In the spectra of other phases, however, the same band is shifted towards shorter wavelengths. The nature of this band is determined by the <sup>6</sup>A<sub>1</sub> → <sup>4</sup>E, <sup>4</sup>A<sub>1</sub> and <sup>6</sup>A<sub>1</sub> + <sup>4</sup>A<sub>1</sub>, <sup>4</sup>E transitions of octahedrally bound Fe<sup>3+</sup> [7]. A well-defined band at 360-380 nm corresponds to the <sup>6</sup>A<sub>1</sub> → <sup>4</sup>E(<sup>4</sup>D) transition. Note that the <sup>6</sup>A<sub>1</sub> → <sup>4</sup>T<sub>1</sub>(<sup>4</sup>D) transition has the same energy.

Table 2. Analysis of the luminescence spectrum of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub>

OUR RESULTS		PUBLICATIONS										
Luminescence of Ni <sub>1-x</sub> Zn <sub>x</sub> Fe <sub>2</sub> O <sub>4</sub>		Luminescence, nm (eV)					Optical absorption, nm					
		ZnFe <sub>2</sub> O <sub>4</sub> [26]	NiFe <sub>2</sub> O <sub>4</sub> [2, 27]	Ni:ZnO [28]	NiO [29]	Fe <sub>3</sub> O <sub>4</sub> [1]	Fe <sup>3+</sup> [3]	Fe <sup>2+</sup> and Fe <sup>3+</sup> [4]	Ni <sup>2+</sup> [5]			
nm	eV											
332	3.734						330 nm 3.756 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> E(G)		330 nm 3.756 eV	<sup>3</sup> A <sub>2g</sub> (F) → <sup>1</sup> T <sub>2g</sub> (G)	
373	3.323	365 nm 3.396 eV	372 nm 3.332 eV [27]		370 nm 3.350 eV				372 nm 3.332 eV	<sup>5</sup> E → <sup>3</sup> E of <sup>T</sup> Fe <sup>2+</sup>		
381	3.254				381 nm 3.254 eV		385 nm 3.220 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> A <sub>1g</sub> (G)	385 nm 3.220 eV	<sup>5</sup> E → <sup>3</sup> T <sub>2</sub> , <sup>3</sup> T <sub>1</sub> of <sup>T</sup> Fe <sup>2+</sup>	385 nm 3.220 eV	<sup>3</sup> A <sub>2g</sub> (F) → <sup>1</sup> T <sub>1g</sub> (G)
396	3.130	401 nm 3.091 eV			396 nm 3.130 eV	400 nm 3.10 eV			402 nm 3.084 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> E <sub>g</sub> (4D) of <sup>O</sup> Fe <sup>3+</sup>	400 nm 3.10 eV	<sup>3</sup> A <sub>2g</sub> (F) → <sup>3</sup> T <sub>1g</sub> (P)
406	3.053				410 nm 3.024 eV		410 nm 3.024 eV					
421	2.896	428 nm 2.896 eV	428 nm 2.896 eV [2]									
448	2.767	454 nm 2.739 eV	441 nm 2.811 eV [2]				440 nm 2.817 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> (G)				
462	2.683	468 nm 2.649 eV	459 nm, 465 nm 2.70 eV, 2.67 eV [3]						459 nm 2.70 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> A <sub>1g</sub> , <sup>4</sup> E <sub>g</sub> transition of <sup>M</sup> Fe <sup>3+</sup>	464 nm 2.672 eV	<sup>3</sup> A <sub>2g</sub> (F) → <sup>1</sup> T <sub>1g</sub> (D)
486	2.551		486 nm 2.551 eV [27]		481 nm 2.577 eV				477 nm 2.599 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> A <sub>1g</sub> , <sup>4</sup> E <sub>g</sub> transition of <sup>O</sup> Fe <sup>3+</sup>		
496	2.499	494 nm 2.509 eV	496 nm 2.499 eV [2]		490 nm 2.53 eV							
507	2.445		502 nm 2.469 eV [2]		507 nm 2.445 eV							
521	2.379		530 nm 2.339 eV [27]				525 nm 2.361 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g</sub> (G)				
545	2.275	539 nm 2.30 eV	535 nm 2.317 eV [2]									
559	2.218								555 nm 2.233 eV	<sup>5</sup> E → <sup>3</sup> T <sub>2</sub> of <sup>T</sup> Fe <sup>2+</sup>		
566	2.194			565 nm 2.194 eV		565 nm 2.194 eV						
571	2.171											
597	2.076	593 nm 2.09 eV	594 nm 2.087 eV [2]						588 nm 2.108 eV	<sup>3</sup> E → <sup>3</sup> T <sub>1</sub> of <sup>T</sup> Fe <sup>2+</sup>		
606	2.046											
627	1.977								631 nm 1.964 eV	<sup>4</sup> A <sub>2</sub> (F) → <sup>4</sup> T <sub>1</sub> (P)		
636	1.949						650 nm 1.907 eV	<sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g</sub> (G)				
673	1.842	665 nm 1.864 eV							670 nm 1.85 eV	<sup>O</sup> Fe <sup>2+</sup> ↔ <sup>O</sup> Fe <sup>3+</sup>		

\* <sup>O</sup>Fe -octahedral and <sup>T</sup>Fe - tetrahedral iron cations

In the absorption spectra, the characteristic energies of the <sup>4</sup>E(<sup>4</sup>D) and <sup>4</sup>T<sub>2</sub>(<sup>4</sup>D) states are separated by about 20 nm. In the spectrum of hematite, however, both transitions appear to be resolved with the <sup>4</sup>E(<sup>4</sup>D) state at 380 nm and the <sup>4</sup>T<sub>2</sub>(<sup>4</sup>D) state at 405 nm. However, one of the transitions is absent in the luminescence spectrum of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> ferrite. Alternatively, we can assume paired excitation either <sup>6</sup>A<sub>1</sub>+<sup>6</sup>A<sub>1</sub>→<sup>4</sup>T<sub>1</sub>(<sup>4</sup>G)+<sup>4</sup>E, <sup>4</sup>A<sub>1</sub>(<sup>4</sup>G); or <sup>6</sup>A<sub>1</sub>+<sup>6</sup>A<sub>1</sub>→<sup>4</sup>T<sub>2</sub>(<sup>4</sup>G)+<sup>4</sup>T<sub>2</sub>(<sup>4</sup>G) transitions. Note that the high-energy, formally independent states of the tetrahedrally and octahedrally coordinated Fe<sup>3+</sup> ligands have the same energies, and naturally, are difficult to distinguish.

Figure 3 shows the luminescence spectra of a thin (40 nm) granular nanofilm Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> (a) from [24] and Fe<sub>3</sub>O<sub>4</sub> (b) from [1]. A red arrow is drawn from the peak of 840 cm<sup>-1</sup> luminescence of Fe<sub>3</sub>O<sub>4</sub> to the maximum of oxygen vacancies, indicating that both of these peaks are of the same nature. The blue color indicates the

components of the luminescence spectrum of ferrite nanopowders, and the red color indicates the maximum at 873 cm<sup>-1</sup> in the Raman spectrum of thin Ni<sub>0.4</sub>Zn<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> films (a).

#### 4. CONCLUSIONS

The PL spectra of Ni<sub>1-x</sub>Zn<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> nanopowders (where x = 0; 0.25; 0.4; 0.5; 0.6; 0.75; 1e) were studied at various energies and excitation powers. The energies of the transitions are determined and a preliminary interpretation is given. The intense luminescence maximum at about 600 nm is the result of dispersionless modes in Ni-Zn ferrites.

#### ACKNOWLEDGEMENTS

This work was supported by the Science Development Foundation, Grants EIF-BGM-3-BRFTF-2+/2017 -15/04/1 and EIF-2013-9(15)-46/05/1.

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