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Studying the Infrared Spectroscopy and Structural Properties of Compounds Fluoride that Blotched by in

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Abstract

In doped calcium fluoride transparent conducting powder were prepared by solid-state reaction method. Structural properties of the samples were investigated as a function of various In-doping levels (x=0.00-0.01-0.02-0.03). The results of x-ray diffraction have shown that the samples are polycrystalline structure in cubic phase and show presence (111), (220), (311), (400) planes in pure CaF2 sample and the preferred orientation is (220) for pure CaF2 and we have peaks correspond (101), (112), (200), (202) for In for all samples and the preferred orientation is (112) for In for all samples. The average of crystallite size is within the range [2.976-0.845nm] for all samples. The relative intensities, distance between crystalline planes (d), crystallite size (D) and lattice parameters (a) were determined. Measuring the infrared spectrum of pure Calcium fluoride, it is found that there are six vibratory frequencies, namely: (3444.52 - 1517.59 - 1138.40 - 876.40 - 639.93 - 436.29) cm- 1 and six vibratory frequencies for indium doped calcium fluoride at 3wt%. (3441.94 - 1516.77 - 1138.44 - 876.55 - 642.09 - 436.44) cm- 1 The study showed that the absorbance values, the absorption coefficient, the refractive index and the optical length were in the indium doped sample by 3wt% greater than that of the pure Calcium fluoride compound and are respectively: α=14.163 cm-1 n=1.767.

Keywords: powder; Calcium fluoride; solid state reaction; Structural properties; Infrared spectrum; rare-earth ions; Optical conductivity

Introduction

Calcium fluoride is represented by the chemical formula CaF2, where CaF2 is an abbreviation for calcium fluoride. Calcium and fluoride ions are the constituent parts of this chemical. It is a crystalline solid that is noted for its transparency and has broad application as a material for transparent windows. This property allows it to be observed in the infrared section of the electromagnetic spectrum. In addition, it is utilized in the production of optical components such lenses, fiber optics, and other optical components. This is another application for the material [1, 2].

The term In-doped calcium fluoride" refers to a specific variety of the mineral calcium fluoride. During the doping procedure, this particular type of calcium fluoride was on purpose adulterated with minute amounts of indium (In). Doping is a procedure that is regularly used to change the properties of a substance, and it is most commonly employed in the field of materials science. In the case of calcium fluoride that has been doped with antimony, the material may become more transparent and more resistant to being harmed by UV radiation as a result of the addition of antimony. In addition, the material may be able to withstand higher doses of ultraviolet radiation. It is also possible for it to raise the material's refractive index, which makes the material more suitable for use in lenses and other optical components. Increasing the substance's refractive index.

Antimony doping is a process in which small amounts of indium (In) are added to a material, such as calcium fluoride (CaF2), to modify its properties. In the case of calcium fluoride, indium doping can change the material's electrical conductivity and refractive index, making it more suitable for use in certain applications.

One common application for indium -doped calcium fluoride is in the production of infrared optics, such as lenses and windows. The addition of indium can improve the transmission of infrared light through the material, making it more efficient for use in these types of devices [4,5]. Indium doping can also be used to improve the performance of calcium fluoride in other applications, such as in the production of coatings for solar panels or as a component in lasers. The specific effects of antimony doping on the properties of calcium fluoride will depend on the amount of antimony added and the processing conditions used.

Calcium fluoride that has been doped with sb has a lot of opportunities for potential uses in a wide range of industries, including the optical, electrical, and healthcare industries. Both infrared spectroscopy and infrared imaging systems typically use it as a window material and as a component of those systems, respectively. Infrared spectroscopy is a subfield of infrared imaging. In addition to that, it is utilized in the production of laser crystals and as a material for the coating of high-quality mirrors. Additionally, it is a substance that is utilized in the production of laser crystals. Research has been conducted in the realm of medicine on In--doped calcium fluoride due to the likelihood that it could be used in the development of fresh new cancer treatments [6, 7].

Calcium fluoride (CaF2) density is 3.18 (g/cm3) melting at 1633 (K) and crystalize in cubic structure with lattice constants a = 5.432 Å. CaF2 is presently the fastest known scintillator. It has an emission component with sub nanosecond decay time. CaF2 has several scintillation emission bands. The fast scintillation light is emitted in the UV bands centered at 220 and 200 nm.

The decay time of the fast component varies between 600 and 800 ps [3]. CaF2 has attracted much attention because of its wide range of potential applications in optoelectronic and microelectronic devices [8, 9]. CaF2 compounds doped with rare-earth ions have been reported to display unique luminescence properties and can thus be used as scintillators [10, 11]. In general, In-doped calcium fluoride is a versatile material that has a range of prospective applications in many different kinds of businesses. These applications may be found in a number of different industries. These applications can be discovered in a wide number of subcultures and subfields. Its one-of-a-kind properties make it a strong contender for use in a wide variety of optical and electronic devices, making it a good option for this kind of application. This study is aimed at characterizing CaF2 compounds doped with rare-earth ions such as in.

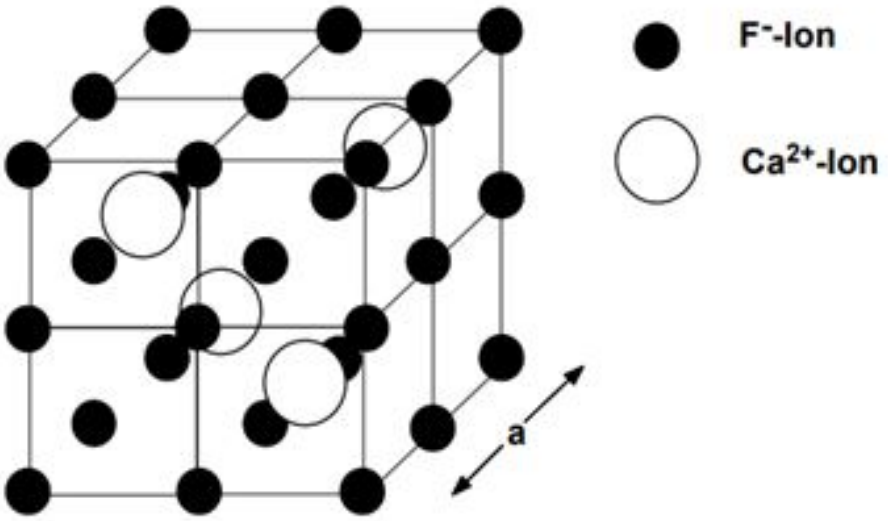


Figure1: The crystalline structure of pure Calcium fluoride

Research Materials and Methods

The following materials have been used in preparing the samples: calcium fluoride CaF2 (99% purity, TITAN BIOTECH LTD, origin India). Indium In (purity 99%, TITAN BIOTECH LTD, origin India).

Devices and Tools Used

Sensitive scale type (SARTORIUS) with an accuracy of (10 - 4) gr is available in the Faculty of Science - Physics Department.

Small agate mortar.

High temperature thermal Oven (1200°C) with a Temperature Regulator.

Preparing the Samples

The samples are prepared by the solid-state reaction method. Accordingly the weights of the powders required for each sample are mixed and calculated using the molecular weight method in order to obtain the compounds required for the study where CaF2 1-x In x; (x=0.0-0.01-0.02-0.03). Then grinding these materials in the agate mortar perfectly well to make the mixture homogeneous and sifting it with a sieve of 90 μm. Then it is put it in a container and we add distilled water to increase the mixing process and homogeneity of the powder. Then we put it on a heater for 3 hours at a temperature of 100° C and the mixing and homogeneity process of the powder occurs by stirring. After that, the powder is placed on a heater with direct contact with the air, then the water evaporates and then we perform a preliminary roasting process inside the oven (pre-sinter) to increase the degree of homogeneity of the mixture. We fix the oven temperature at 700°C for three hours, then we turn off the oven, which means to stop the roasting process and leave the sample inside the oven until it cools and reaches room temperature, thus we get rid of impurities that evaporate at high temperatures. Then we grind the powder resulting from the roasting process in its first stage. Then we perform the second roasting process where we fix the oven temperature at 100° C for an hour and then we raise the temperature 50°C every 15min until we reach the temperature of 700°C where we fix the oven temperature at it for 3 hours in order to get the crystal structure in its correct form.

Results and Discussion

Infrared Spectroscopy

The IR spectrum of pure calcium fluoride and indium doped calcium fluoride was measured using the spectrometer asco type FT / IR-460 plus available in the central laboratory of the Faculty of Science - aleppo University, working in the range [400 - 4000] Cm-1. Where the permeability T was measured by the frequency function υ , the absorbance A, the absorption coefficient α, the damping factor K, the refractive index n and optical conductivity σopt were calculated:

Permeability T

It is defined as the ratio between the intensity of the penetrating radiation to the intensity of the fallen radiation, it has been taken from the device itself and then by using the appropriate mathematical equations, other optical parameters have been calculated.

Absorbency A

It is the ratio between the intensity of the absorbed radiation and the intensity of the fallen radiation, calculated from the equation [15]:

$$A = \log\left(\frac{100}{T\%}\right) = \log\left(\frac{1}{T}\right)$$

T represents Permeability.

Absorption coefficient α

defined as the ratio between the decrease in the flow of the fallen radiation energy to the unit of distance towards the spread of the fallen light wave within the field, and is calculated from the equation:

$$\alpha = 2.303\frac{A}{d}$$

A represents absorbency, d = 1mm the thickness of the material

The damping factor k

is defined as the amount of energy absorbed by the electrons of the studied material from the energy of the radiation photons that fall on it, and is calculated from the equation [17]:

$$K = \frac{\alpha}{4\pi v}$$

Refractive Index N

which is the ratio between the speed of light in the vacuum to its speed in the field, and it is calculated from the equation [18]:

$$n = \left(\frac{100 - T\%}{T\%}\right)^{\frac{1}{2}}$$

Optical length L

The inversion of the absorption coefficient [16]:

$$L = \frac{1}{\alpha}$$

Optical conductivity σ_{opt}

Optical conductivity is related to the refractive index and the damping factor according to the following equation [18]:

$$\sigma_{opt} = \frac{1}{30} nkv$$

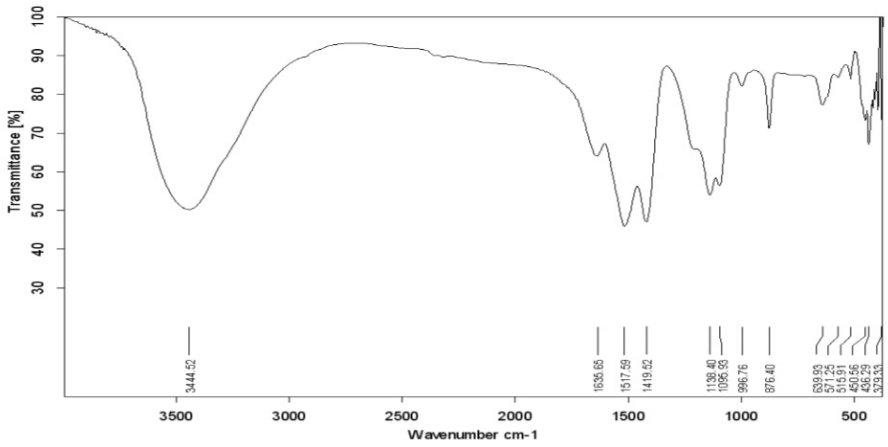


Figure2: Represents the FTIR spectrum for pure calcium fluoride

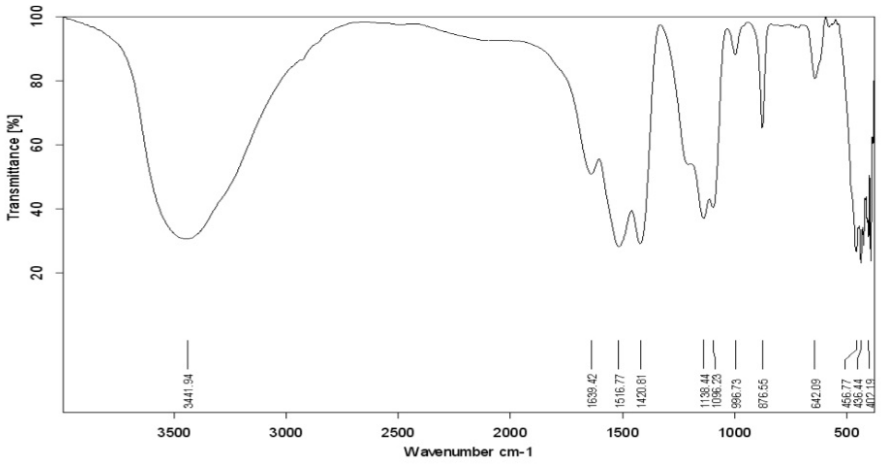


Figure3: shows the FTIR spectrum of In doped CaF2 samples (3wt%).

FTIR analysis of samples has been performed at room temperature within the range [400-4000] Cm-1. Some bundles appeared within this range, including the absorption bundle at the top corresponding to the number of vibratory frequency 1516.77 cm-1, 3441.94 cm-1 which belongs to the vibratory pattern of the hydroxyl group (vibration of hydroxyl group). This is due to the absorption of water vibration, the vibratory pattern also shows stretching and retraction (stretching vibrational mode) belonging to the O-H group [12]. The location of the absorbent bundles and peaks also depends on the crystalline structure of the material and its chemical composition, as well as on the morphology of the material [13].

Pure calcium fluoride is characterized by a set of vibrational frequencies within the range [400-4000] Cm-1 which is: (3444.52 – 1517.59 – 1138.40 – 876.40 – 639.93– 436.29) cm- 1.

indium doped calcium fluoride by 3wt% is characterized by a set of vibrational frequencies within the range [400-4000] Cm-1 which is:

(3441.94 - 1516.77 - 1138.44 - 876.55- 642.09 - 436.44) cm- 1

An absorbent value has been observed at about the frequency 1138.44 cm-1 belonging to the C-H group, and this could be due to adsorption and interaction between carbon dioxide atoms with water during the sintering process [14].

From the permeability spectrum by the frequency function indicated in Figure (3), the physical quantities of pure calcium fluoride have been calculated, which are the absorbance, the absorption coefficient, the damping coefficient, the refractive index, the wavelength, the optical conductivity, which are shown in Table (1).

Table1: shows the vibrations frequency of pure calcium fluoride with corresponding permeability values for each frequency, absorbance and absorption coefficient, damping factor, refractive index, optical length and optical conductivity.

$\upsilon(cm)^{-1}$	T%	A	$\alpha(cm)^{-1}$	n	$k\times 10^{-4}$	L(cm)	$\sigma_{(opt)}(\Omega cm)^{-1}$
3444.52	50.006	0.283	6.517	0.999	1.506	0.153	0.017
1517.59	46.022	0.337	7.761	1.082	4.071	0.128	0.022
1138.40	50.082	0.300	6.909	0.998	4.832	0.144	0.018
876.40	69.825	0.155	3.569	0.657	3.242	0.280	0.006
639.93	78.265	0.106	2.441	0.526	3.037	0.409	0.003
436.29	67.128	0.173	3.984	0.699	7.271	0.251	0.007

Table2: shows the vibrations frequency of the indium doped calcium fluoride by (3wt%) with corresponding permeability values for each frequency, absorbance and absorption coefficient, damping factor, refractive index, optical length and optical conductivity.

$\upsilon(cm)^{-1}$	T%	A	$\alpha(cm)^{-1}$	n	$k\times 10^{-4}$	L(cm)	$\sigma_{(opt)}(\Omega cm)^{-1}$
3441.94	31.248	0.505	11.630	1.483	2.690	0.085	0.045
1516.77	30.0885	0.521	11.998	1.524	6.298	0.083	0.048
1138.44	39.128	0.407	9.373	1.247	6.555	0.106	0.031
876.55	70.645	0.150	3.454	0.644	3.137	0.289	0.005
642.09	82.080	0.086	1.957	0.467	2.426	0.510	0.002
436.44	24.246	0.615	14.163	1.767	25.840	0.070	0.066

The absorption coefficients variations are drawn by the function of the vibrational frequency of pure calcium fluoride and the indium doped calcium fluoride as shown in Figure (4).

Figure (4) shows the change in the absorption coefficient of pure and indium doped calcium fluoride, we note that the largest absorption coefficient value was (7.761cm-1) corresponding to the frequency (1517.59 cm-1) cm-1 and the smallest absorption coefficient value was (2.441cm-1) corresponding to the frequency (639.93cm-1), and the largest value of absorption coefficient to the In doped calcium fluoride was (14.163cm-1) corresponding to the frequency (436.44cm-1) and the smallest value of absorption coefficient was 1.957cm-1 corresponding to the frequency (642.09cm-1).

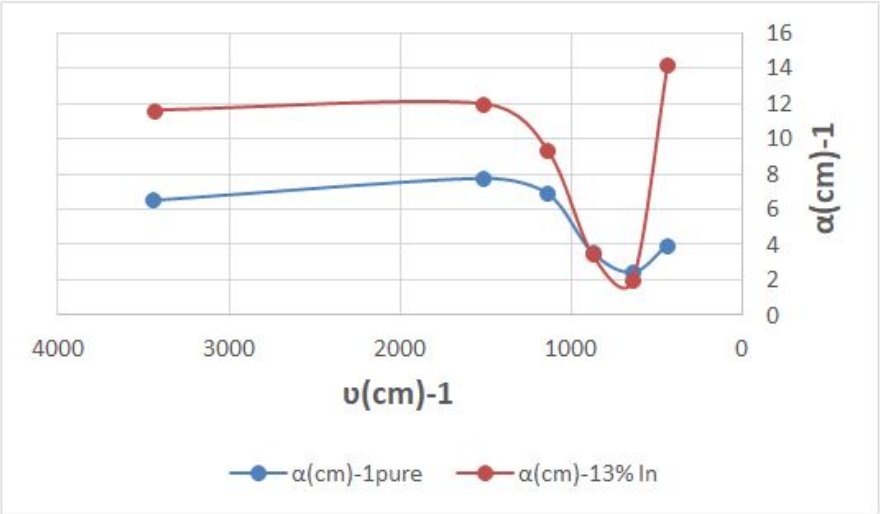


Figure4: shows the absorption coefficient variations by the function of the vibrational frequency of pure calcium fluoride and the indium doped calcium fluoride by (3wt%).

The optical conductivity variations are also drawn by the frequency function of pure calcium fluoride and the indium doped calcium fluoride, as shown in Figure (5).

Figure (5) shows the change in the conductivity of pure and indium doped calcium fluoride, we note that the largest value of the conductivity was (0.022 Ωcm-1) corresponding to the frequency (1517.59 cm-1) cm-1 and the smallest value of conductivity was (0.003 Ωcm-1) corresponding to the frequency (639.93cm-1), and the largest value of the conductivity to the In doped calcium fluoride was (0.066 (Ωcm)-1) corresponding to the frequency (436.44cm-1) and the smallest value of conductivity was (0.002 Ωcm-1) corresponding to the frequency (642.09cm-1).

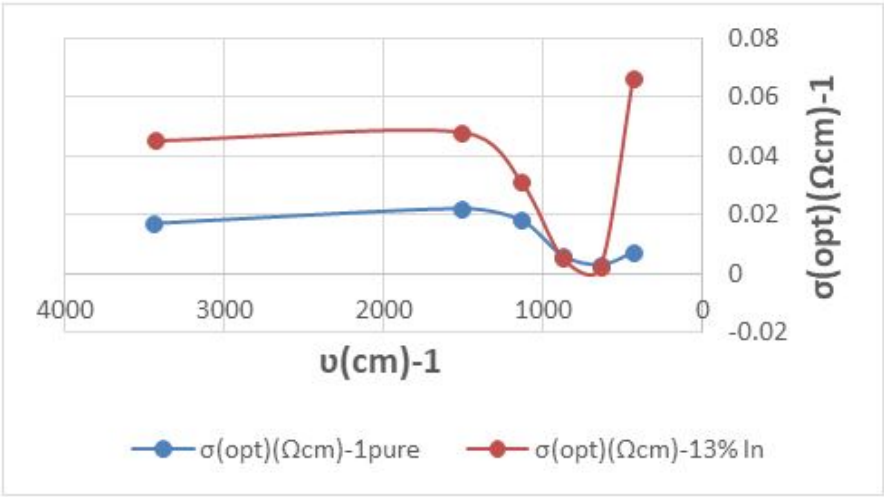


Figure5: shows the studying of the conductivity variations by the frequency function of pure calcium fluoride and the indium doped calcium fluoride by (3wt%).

Figure (6) shows the change in the absorbance of pure and indium doped calcium fluoride, we note that the largest absorbance value was (0.337) corresponding to the frequency (1517.59cm-1) and the smallest value of absorption was (0.106) corresponding to the frequency (639.93cm-1), and the largest value of absorbance corresponds to the In doped calcium fluoride was (0.615) corresponding to the frequency (436.44cm-1) and the smallest value of absorbance was (0.086) corresponding to the frequency (642.09cm-1).

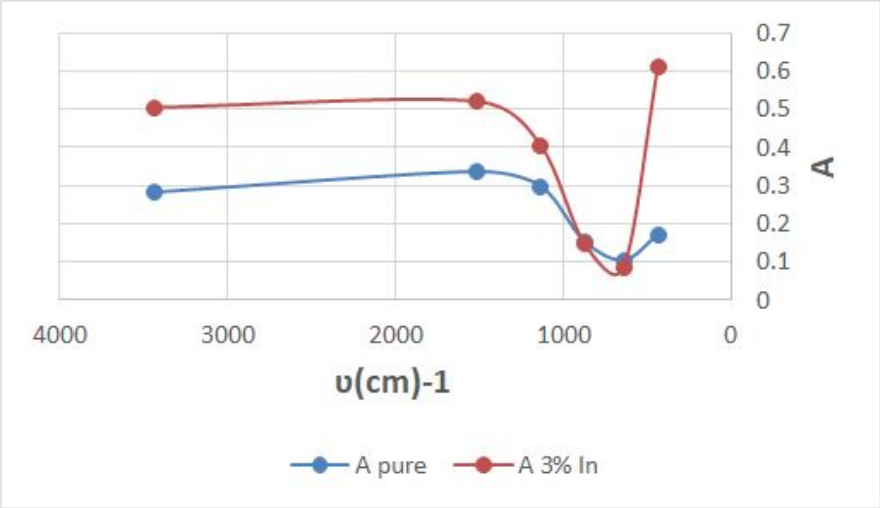


Figure6: Absorbance variations by the frequency function of pure calcium fluoride powder and the indium doped calcium fluoride by (3wt%).

Structural Properties

Device type XRD-PW 1840 PHILIPS production is connected to a computer with software for diffraction spectrum processing.

The X-ray diffraction patterns of undoped and In doped CaF2 powders prepared with various In concentration 0wt%, 1wt%, 2wt% and 3wt% are shown in Figure (7).

The XRD reveals that all samples are having polycrystalline nature with cubic structure.

The relative intensities of undoped and In doped CaF2 powders are calculated. The distance between crystalline planes values (d) are calculated by using following relation:

$$2d.\sin\theta = n\lambda$$

Where d is distance between crystalline planes (A°), θ is the Bragg angle, λ is the wavelength of X-rays

$$(\lambda = 1.78897A^{o})$$

The crystallite size is calculated from Scherrer's equation [20]:

$$D = \frac{0.94\lambda}{\beta\cos\theta}$$

Where, D is the crystallite size, λ is the wavelength of X-ray, ß is full width at half maximum (FWHM) intensity in radians and θ is Braggs's angle.

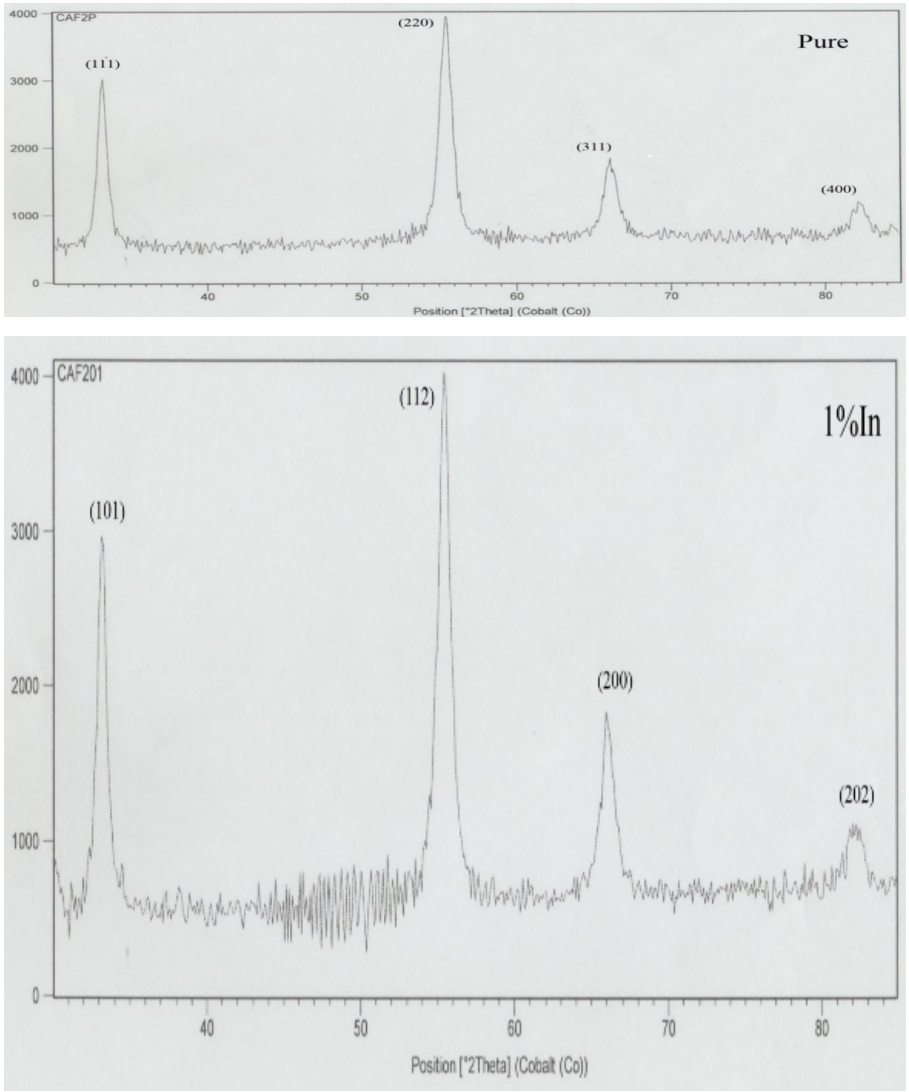
The dislocation density is defined as the length of dislocation lines per unit volume and calculated by following equation [21]:

$$\delta = \frac{1}{D^2}$$

The lattice constants a for cubic phase structure is determined by the relation [22]:

$$a = d\sqrt{h^2 + k^2 + l^2}$$

Where d and (hkl) are distance between crystalline planes and Miller indices, respectively.



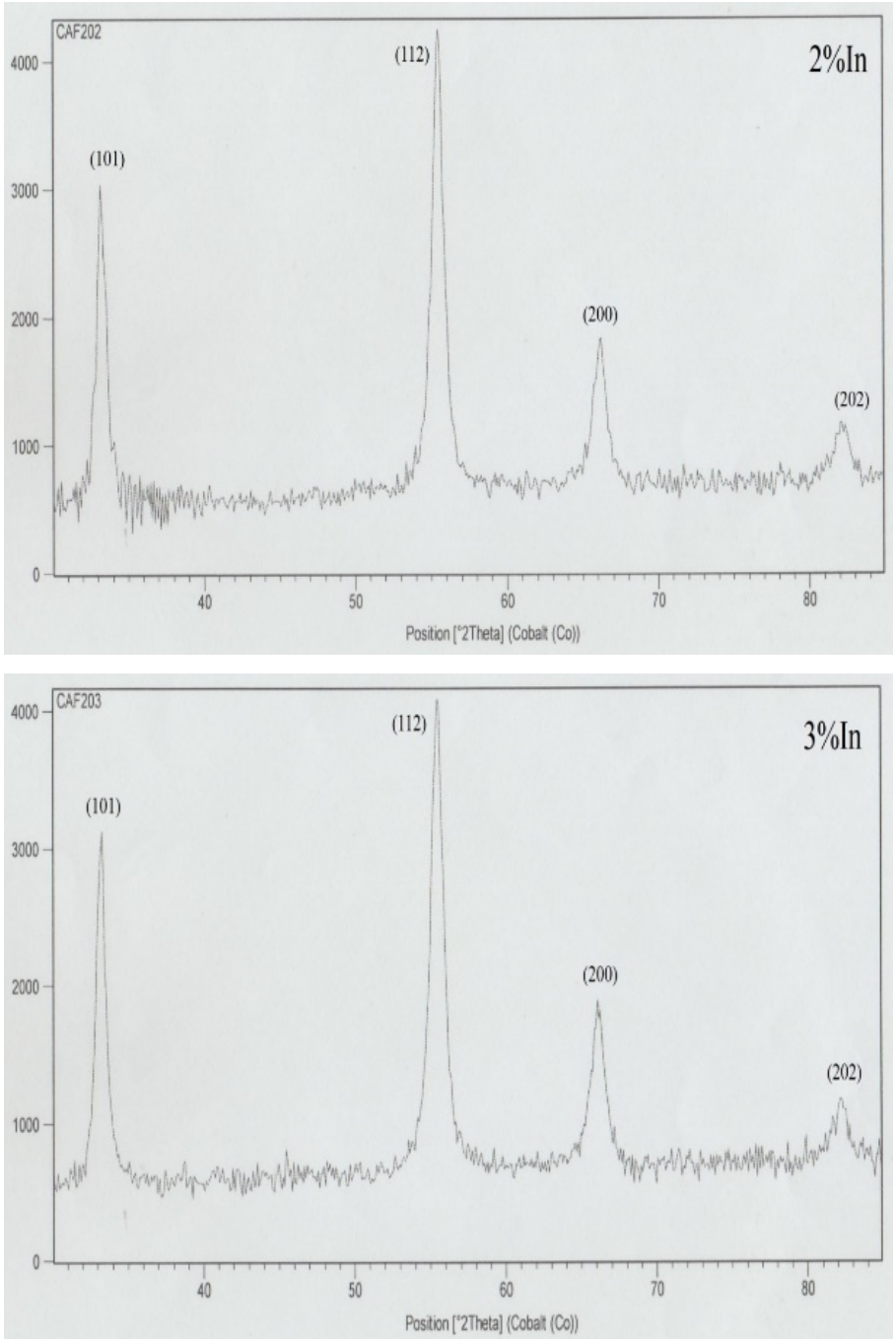


Figure7: XRD results of pure CaF2, 1wt% In doped CaF2, 2wt% In doped CaF2, 3wt% In doped CaF2.

Table3: shows results of structural values of undoped CaF2 sample.

Samples	2θ (deg)	(hkl)	d (A°)	Rel. int. [%]	β (deg)	D (nm)	Average D(nm)	Δ	Lattice const.a(Å)
								10^{15} line/m^2	
CaF_2	33.12	(111)	3.138	80	1.250	1.403	1.504	508.024	5.456
	55.23	(220)	1.929	100	1.720	1.103		821.956	
Pure	65.78	(311)	1.647	48	1.350	1.483		454.692	
	82.23	(400)	1.360	33	1.100	2.029		242.904	

Table4: shows results of structural values of In doped CaF2 samples 1wt%.

Samples	2θ (deg)	(hkl)	d (A°)	Rel. int. [%]	β (deg)	D (nm)	Average D(nm)	Δ 1015line/m2	Lattice const. a(Å)
CaF2: In (1wt%)	33.08	-101	3.144	72	1.355	1.294	1.608	597.216	4.747
	55.01	-112	1.938	100	1.75	1.083		852.595	
	65.52	-200	1.653	43	1.85	1.08		857.338	
	82.24	-202	1.36	29	0.75	2.976		112.91	

Table5: shows results of structural values of In doped CaF2 samples 2wt%.

Samples	2θ (deg)	(hkl)	d (A°)	Rel. int. [%]	β (deg)	D (nm)	Average D(nm)	Δ 1015line/m2	Lattice const. a(Å)
CaF2:In (2wt%)	33.51	-101	3.105	71	1.325	1.325	1.474	569.597	4.685
	55.78	-112	1.913	100	2.25	0.845		1400.511	
	66.21	-200	1.638	42	1.25	1.606		387.711	
	82.03	-202	1.363	30	1.05	2.122		222.079	

Table6: shows results of structural values of in doped CaF2 samples 3wt%.

Samples	2θ (deg)	(hkl)	d (A°)	Rel. int. [%]	β (deg)	D (nm)	Average D(nm)	Δ 1015line/m2	Lattice const. a(Å)
CaF2:In (3wt%)	33.71	-101	3.089	78	1.65	1.064	1.47	883.317	4.68
	55.82	-112	1.911	100	1.75	1.087		846.332	
	66.06	-200	1.641	42	1.45	1.383		522.824	
	82.08	-202	1.362	30	0.95	2.346		181.695	

Conclusions

1. The pure calcium fluoride FTIR spectrum has shown some vibrational frequencies within the range [400-4000] Cm-1 which are:

(3444.52 - 1517.59 - 1138.40 - 876.40 - 639.93 - 436.29) cm- 1

The FTIR spectrum of the indium doped calcium fluoride by 3wt% showed vibrational frequencies within the range [400-4000] Cm-1, the most notably are:

(3441.94 - 1516.77 - 1138.44 - 876.55 - 642.09 - 436.44) cm- 1.

2. The absorbance value for the pure sample varies within the range [0.106 - 0.337], and for the doped sample, the absorbance value varies within the range [0.086 - 0.615].

3. The absorption coefficient value varies within the range of the pure sample [2.441 - 7.761] Cm-1

4. The value of the refractive index for the pure sample varies within the range [0.526 - 1.082].

5. The value of the optical length L concerning the pure sample varies in the range [0.128 - 0,409] cm, and for the doped sample the value of the optical length varies within the range [0.070 - 0.510] cm.

6. The optical conductivity value σopt for the pure sample varies within the range [0.003 - 0.022] (Ωcm)-1, for the doped sample, the value of the optical conductivity varies in the range [0.002 - 0.066] (Ωcm)-1.

7. The largest values of absorbance, absorption coefficient, refractive index and optical length were greater in the indium doped sample by 3wt% for the vibrational frequency 436.44 cm-1.

8. The largest values of the absorbance, the refractive index, and the absorption coefficient were in the pure sample for the frequency 1517.59cm-1.

9. X-ray diffraction patterns confirm that the samples have polycrystalline nature with cubic structure and show presence (111), (220), (311), (400) planes in pure CaF2 sample. The preferred orientation is (220) for pure CaF2.

10. For the doped samples we have peaks correspond to (101), (112), (200), (202). The preferred orientation is (112).

11. The average of crystallite size is within the range [2.976-0.845nm] for all samples. It was defined that the lattice constants a for all the samples, were almost identical with JCPDS values.

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