Thin-film structures with nanocrystals: an origin of enhanced photo-response

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Abstract. To discover well the properties of nanothin crystalline layers and nanometer-sized crystals, we investigated the relatively thick multilayer structures composed of high quantity of nanothin layers with nanocrystals. Alternate nanolayers of 150–10 nm thicknesses with LiF, CaF2 and CdS nanocrystals have been deposited onto irradiation-resistive substrates by thermal evaporation of pure crystals. Some multilayers were γ-irradiated in air at room temperature with dose of 83 kGy. X-ray diffraction and microscopy studies reveal that the multilayers consist of nanocrystals with cubic phase and defined size. Thin-film structures were oriented along the (111) plane. Absorption spectra of non-irradiated LiF nanocrystals of 100 nm size suggest evidence of metal colloids presence. We find that photoluminescence spectra of γ-irradiated nanostructures with metal colloids and various LiF contents show the enhancement of F3+-colour centres excitation in the region of metal colloids absorption and the increase is observed of the emission intensities ratio of F3+ and F2 centers with respect to initial crystals γ-coloured in identical conditions. Emission intensities of both centers under excitation at 458 nm correlate with LiF content. These effects, which are related to high-quality nanostructures, but at the same time depend strongly on the defect content, especially as far as their 1–2 ps nonlinearities are concerned, could depend on nanocrystal purity and metal excess collection in their boundaries regions. Our results provide an original contribution to the understanding of the influence of the nanolayer-by-nanolayer deposition, γ-irradiation on these specific structures, and of the metal aggregates on the properties of nanocrystals and nanolayers.

1. Introduction
Nano-sized layers, films, crystals and multilayers play an important role in future technology as they exhibit different and often unique properties with respect to the initial macroscopic materials [1–6]. Film structures with nanocrystals have attracted much interest for the possibility to grow them in their final position within switching devices, detectors, efficient dosimeters, emitters and solar cells. The possibility of using these nanostructures not only for fundamental studies, but also for novel devices fabrication, is stimulating the efforts to control the nanocrystal size, shape, structure, space arranging as well as their composition and structural defects. Unfortunately, not only bulk, but boundary (surface) states of nanolayers and nanocrystals affect strongly their properties as their size reduces [1, 7]. The properties of the nanoelements as prepared and post-growth processed by different methods

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can be affected by the presence of various defects and also by aggregation of impurities and intrinsic defects to the colloids in the bulk and surface regions related to the used preparation technique. Several physical deposition methods are usable for preparation of thin films and nanostructures such as laser-assisted, electron-beam or thermal evaporation from composite targets and undoped bulks, sequential sputtering or evaporation [1–6, 8]. Ion implantation [9], thermal treatments [10] and ionising radiation bombardment (combined with special annealing [11–14]) are established tools for metal nanoparticles formation in solids. Understanding the structural evolution during the preparation of nanocomposites can be of great help in understanding how to control the characteristics of the final products readily.

Lithium fluoride is a well-known material which, after irradiation by ionizing radiation (electrons, ions, γ-rays and X-rays) or laser light, is characterized by F-colour centres (CCs) aggregates [3–5] and metal colloids formation sensitive to the content of oxygen, hydroxyl and divalent metal ions in initial crystals [11–14]. Clusters of F-CCs (lithium colloids) and impurity colloids can be created in the irradiated regions of LiF and at temperatures where diffusion of the defects is possible [10–12]. Even though the actual mechanism of the formation of such colloids is still an open question, a lot of attention was devoted to study optical properties of the crystals and their near surface nanothin layers containing these metallic aggregates. Since thin-film nanolayers and nanocrystals have been studied only in a limited way, in this paper, we carried out the following measurements to investigate them.

2. Experimental details and measurements
Microstructural and optical characteristics of the investigated samples are summarized in figures 1–8. The experimental samples are multilayers and thin-film interferometers (TFIs) with the intermediate multilayers [15–17]. Each structure unit is a thin-film nanolayer with a nanocrystal array inside it (figures 1, 2). The geometry of the nanostructures can be tuned by controlling the thicknesses and the compositions of the “sensitizing” and complementary, buffer, nanolayers. Every nanolayer can be designed of one material [15] and as composition of nanocrystals, clusters of two materials [16].

Figure 1. Schematic representation of the periodic multilayer nanostructures with c-oriented nanocrystals prepared from one (a), two (b) and more than two initial materials (c).

2.1. Samples preparation
The elaborated samples consist of dielectric (LiF, CaF₂) or/and semiconductor (CdS) nanocrystals. Samples with various “sensitizing” nanocrystals are grown on radiation-resistant, flat substrates in the layer-by-layer manner of the reproducing of thin-film nanolayers with defining compositions [15, 16]. “Sensitizing” nanolayers are prepared with thicknesses equal to nanocrystal sizes [15], and by using special techniques for nanocrystal ordering [17]. The approach presented in this paper can be extended to fabricate thin-film multilayer structures of a wide range of materials. To avoid radiative coloration, thermal evaporation was the preferred fabrication technique. Undoped crystals, 99.999% pure, are used as starting materials. The clean substrates were kept at room temperature (RT) on a copper
holder, and the vacuum pressure in the evaporation chamber was about \(10^{-6}\) Torr. The films thicknesses were measured by interference method. As-prepared (fresh) samples are kept for seven days in a dry box before starting the optical measurements in the open atmosphere at RT at about 50% humidity. Crystals and films produced in the manner of continuous growth of the result thicknesses were used as reference samples. After the growth some samples were \(\gamma\)-irradiated at RT up to 83 kGy [19]. Self-assembled nanolayers of LiF and CaF\(_2\) nanocrystals were used as templates. In the case of CdSe/CaF\(_2\), CdS/CaF\(_2\) [20] and LiF/CaF\(_2\) nanostructures (figure 2), more radiation-resistant crystalline CaF\(_2\) nanolayers were used to promote the \(c\)-axis ordering and the size restriction of “sensitizing” CdSe, CdS and LiF nanocrystals. After thermal evaporation and post-growth \(\gamma\)-colouration, spatially ordered arrays of CCs could be fabricated in the periodic multilayer nanostructures (figure 1b) [6].

2.2. Samples characterization
The microstructure of thin-film samples is determined by X-ray phase analysis, scanning electron microscopy (SEM) and atomic force microscopy (AFM). The \(c\)-axis ordering (texture) analysis of thin-film nanostructures was performed by means of pole figures and \(\theta\)-2\(\theta\) diffraction patterns (figures 2–6). A Seifert XRD 3003 PTS four circles diffractometer employing CuK\(_\alpha\) (\(\lambda = 1.54059\) Å) radiation was used for pole figure measurements. The primary beam was collimated by a 0.5 mm pinhole and a 1 mm slit was placed in front of the detector. The accelerating voltage and current was fixed at 40 kV and 30 mA, respectively. Pole figures were recorded varying \(\phi\) between 0° and 360° with a sampling interval of 4°, and varying \(\chi\) between 0° and 75° with \(\Delta\chi = 2.5°\). The acquisition time was 5 seconds. X-ray \(\theta\)-2\(\theta\) diffraction pattern were collected by using a Rigaku Geigerflex diffractometer with CuK\(_\alpha\) radiation. The diffractometer was equipped with a graphite monochromator in order to suppress K\(_\beta\) radiation and to increase signal to noise ratio. The divergence slit, scattering slit and receiving slit were 0.5°, 0.45° and 0.3°, respectively. The measurements were performed with a sampling interval of 0.05° and a fixed time of 10 seconds.

Optical transmittance (\(T\)) and reflectance (\(R\)) spectra of samples are measured with a dual beam Cary 500 Scan and a Lambda 19DM Perkin-Elmer spectrophotometers in the spectral range 190–3300 nm (figures 6, 8). Optical absorption (OA) (figure 7) is derived from transmittance and is used to estimate the defects in nanocrystals with respect to initial crystals. The photoluminescence (PL) measurements were carried out by using a Jobin Yvon Fluorolog-3 spectrofluorometer with the front-face detecting geometry, by exciting the samples at the wavelength of 458 nm (M-band, LiF), see figures 7b,c. Nonlinear properties were detected using a pump-and-probe technique and 3-ps [11–15] and 150-fs laser pulses [3, 5], and reported in figure 8. All measurements were performed at RT.
3. Thin-film structures with dielectric nanocrystals

3.1. Thin-film structures with nanocrystals of one fluoride material

X-ray diffraction (XRD)-, SEM- and AFM-results show that non-irradiated multilayers are structures with definite size of nanocrystals. (Final thickness of LiF, CaF$_2$ and LiF/CaF$_2$ structures is approximately 6.5 μm.) Presence of (111) and (222) peaks in XRD data of the multilayers indicates that they have c-axis orientation perpendicular to the substrate surfaces. The XRD patterns of LiF and CaF$_2$ multilayers, see figure 2, show intense (111) peaks at 2θ = 38.7º, 28.3º and a considerably less intense (222) peaks at 2θ = 83.1º, 58.5º, respectively. Not-presented here XRD-, SEM- and AFM-results validate the fact that irradiation neither causes a change in the packing nor increases the size of nanocrystals. The (111) peaks prevail in the XRD-patterns of samples, and γ-irradiation with the dose of 83 kGy only improves the crystallinity and the orientation of nanostructures (increase in the intensities of (111) peaks). The XRD results correlate well with the texture analysis (figures 3–5).

![Figure 3](image-url)
In figure 3a two (2d) and three dimensional (3d) (111), (200) and (220) LiF pole figures of the non-irradiated LiF sample are reported. A sharp (111) texture is present. The Full Width at Half Maximum (FWHM) of the pole located at $\chi=0$ in the (111) pole figure is about 6°, and the FWHMs of both (200) and (220) poles are 8°. This is due to a very low mosaic spread in the growth direction. Figure 3b shows a magnification of the (111) pole figure in the range $\chi<30^\circ$. The isointensity lines were chosen in the following way: the intensity of the nth line is $I_n = I_{\text{max}}/2^n$. The circular shape of the pole indicates that the in-plane nanocrystal distribution is uniform. This hypothesis is supported by the analysis of the cut of the (200) pole image at $\chi=55^\circ$, shown in figure 3c: no difference in intensity is observable varying $\phi$ angle, confirming that any preferential in-plane direction does not exist.

The (111), (200) and (220) LiF pole figures for the $\gamma$-irradiated LiF sample are shown in figure 4. For this sample perfect (111) texture is presented (a). The FWHM of the (111), (200) and (220) LiF poles are 5°, 8° and 7°, respectively. The detailed (111) pole in the range $\chi<30^\circ$ (b) and the cut of the (200) pole figure at $\chi=55^\circ$ (c) are also reported. The (111) LiF pole profile is perfectly circular and the (200) pole $\phi$-scan is constant indicating a high in-plane isotropy, differently from what we observed for non-irradiated LiF sample in which a low fraction of slightly misoriented nanocrystals can be inferred.

![Figure 4](image)

**Figure 4.** 2d and 3d (111), (200) and (220) LiF pole figures of the $\gamma$-irradiated LiF nanostructures (a). The perfect texture is shown. Magnification of (111) LiF pole figure, $\chi<30^\circ$ (b) and $\phi$-scan of (200) figure demonstrating in-plane isotropy (c).
It must be emphasized that the difference is very small: an analysis of the (111) LiF pole figures revealed that the integrated intensity of the range $\chi < 10^\circ$ ($I_{\chi<10}$) to the integrated intensity of the total (111) pole figure ($I_{\text{total}}$) ratio varies from 14.95% (for the non-irradiated LiF sample) to 15.05% (for the sample $\gamma$-irradiated at RT with dose 83 kGy). Conversely, the pole maximum is increased of about 20% in the irradiated sample confirming the effect of the structure improving due to the $\gamma$-irradiation.

In figure 5 the (111), (400) and (220) CaF$_2$ pole images of non-irradiated CaF$_2$ sample are reported (a). Also in this case, the presence of a good (111) texture is evident. The FWHM of (111), (400) and (220) poles are 5°, 7° and 7°. The (111) peak profile is circular down to the lowest intensity and no evidence of peak broadening was observed. The pole is misaligned respect to the centre of about 0.5°, as can be seen in figure 5b. Since the measurement was repeated several times, an error in the mounting of the sample can be excluded. A different profile of the $\phi$-scan performed on the (400) and (220) poles was recorded (figure 5c), characterised by a weak intensity dependence vs $\phi$. This behaviour is due to the (111) CaF$_2$ direction not exactly perpendicular to the substrate surface.

![Figure 5](image-url)

**Figure 5.** 2d and 3d (111), (200) and (220) CaF$_2$ pole figures of non-irradiated CaF$_2$ nanostructures (a). Magnification of (111) CaF$_2$ pole figure, $\chi < 30^\circ$ (b). A misalignment of ~0.5° is present and is consistent with the (400), (220) $\phi$-scan (c).

Not reported here the poles for the $\gamma$-irradiated CaF$_2$ sample revealed the development of more strong (111) texture. The FWHM of the (111) pole is about 4° indicating a very small mosaic spread.
Thin-film structures with nanocrystals of various fluoride materials
To establish the influence of content of LiF nanocrystals with given size on PL spectra of multilayers, two LiF/CaF$_2$ structures have been prepared. The first, LiF/CaF$_2$(1) (figure 1b, period 1), is the 48-period structure of 100 nm LiF-nanolayers and 50 nm CaF$_2$-nanolayers. The next, LiF/CaF$_2$(2), is the 24-period structure consisting of 100 nm LiF- and 220 nm CaF$_2$-nanolayers (figure 1b, period 2).

Figure 6. 3d (111) LiF, CaF$_2$ pole images of fresh LiF/CaF$_2$(1) (left) and LiF/CaF$_2$(2) multilayers (middle, right) (a). XRD patterns of LiF/CaF$_2$(2) before (1), after irradiation (2) and fresh LiF/CaF$_2$(1) (3) (b). Transmission spectra of the multilayers and substrate (c). Reflection spectra of LiF/CaF$_2$(1) vs. angles of incidence in the range 20º–50º (d). The graphs are $p$-polarized light.

Both multilayers were composed of ~100 nm LiF and ~50 nm CaF$_2$ nanocrystals and were $c$-oriented structures (figures 6a,b). In figure 6a the (111) LiF and CaF$_2$ pole images of as-prepared LiF/CaF$_2$(1) (left) and LiF/CaF$_2$(2) samples (middle, right) are reported. A well-resolved (111) LiF texture is present on both cases. The quantity of used isolines are 128, and the minimal and maximal intensities in the (111) LiF pole figures are about 91.0 and 283 cps for the LiF/CaF$_2$(1) samples, and 45.0 and 115.4 cps for the LiF/CaF$_2$(2) samples. This is due to the result LiF thickness in the LiF/CaF$_2$(1) sample (4.5 μm) 2.7 time larger with respect to the LiF/CaF$_2$(2) sample (1.7 μm). The CaF$_2$ pole image (figure 6a, right) indicates that the preferential nanocrystal orientation is (111) direction. The crystallinity and (111) texture improving for the $\gamma$-irradiated LiF/CaF$_2$ samples is supported by the XRD patterns shown in figure 6b: strong difference is observable between the data measured for LiF/CaF$_2$(2) before (curve 1) and after $\gamma$-irradiation at RT with dose 83 kGy (curve 2).

High-reflectivity and transmissivity bands are provided by the LiF/CaF$_2$ structures (figure 2c). Spectral positions of the bands were not changed after $\gamma$-irradiation but were sensitive to the polarization state and angle of incidence of light, see figure 2d. The LiF/CaF$_2$ structures, covered at the surface by CaF$_2$ nanolayers, are more mechanically stable and resistant to external influences than LiF nanostructures.
3.3. Optical absorption features of non-irradiated thin-film structures

OA spectra of non-irradiated LiF nanostructures and initial crystals with small content of Mg\(^{2+}\), O\(^{-}\), and OH ions [13] show well-resolved absorption of Mg\(^{2+}\) colloids [11, 12] (figure 7a). (The non-irradiated and \(\gamma\)-irradiated substrates remain non-absorbing in the above spectral range.) According to the literature, besides Mg colloids with the absorption band peaked at 4.4–4.6 eV, the low-wavelength absorption band at 3.6–4.1 eV, related to the formation of more large Mg colloids or intrinsic Li colloids [11, 12], are reliably distinguished in non-irradiated LiF nanostructures (figure 7a) [21] and initial crystals \(\gamma\)-irradiated with various doses and bleached by annealing after irradiation (figure 7a, insert) [13, 14].

![Figure 7](image)

**Figure 7.** OA spectra of non-coloured (a) and PL spectra of \(\gamma\)-coloured nano (bold curve) and bulk (solid curve) crystalline structures (b) and the structures with various LiF contents (c).

3.4. PL spectra of the irradiated thin-film structures

Non-presented here PL spectra of \(\gamma\)-irradiated CaF\(_2\) nanocrystals are composed of the reference 425 nm band excited at the wavelength of 370 nm. The PL spectra of initial LiF crystal, LiF and LiF/CaF\(_2\) nanostructures having the same size of LiF nanocrystals and irradiated in analogous conditions are given in figures 7b,c. In comparison with the initial crystals, an increase of \(F_3^+\)-CCs excitation in the region of Mg colloids absorption in nanostructures is observed, together with an enhanced emission in the green region with respect to the red one for \(\gamma\)-irradiated film structures (figures 7b). The normalized emission spectra of nanostructures with various LiF content (bold) have the same shape: the \(F_3^+\) photoluminescence band intensity at \(\lambda_{\text{max}} = 538\) nm increases of the same factor in comparison with the \(F_2\) emission at 670 nm with respect to the spectrum of the crystal (solid curve). The PL spectra show an increase (correlated with LiF nanocrystal content) of the intensities of \(F_3^+\) and \(F_2\) bands (figure 7c). These results could be explained by the high structural and optical qualities of these nanostructures (the overall emission is proportional to the quantity of luminescent LiF nanocrystals) and by the nanocrystal purity with metal excess collection in their boundaries regions [22].

4. Thin-film structures with semiconductor and dielectric nanocrystals

We measured nonlinear optical (NLO) effects for the reference films (figure 8a, curve 1), elaborated multilayers (figure 8a, curves 2, 3) and specially-elaborated multipeak TFIs, containing \(c\)-oriented cubic CdS nanocrystals of defined sizes, as reported in figure 8. Sample 3 and TFIs intermediate layers are composite film structures with semiconductor (CdS) and dielectric (CaF\(_2\)) nanocrystals (figure 1c). Separation of the transient shift and absorption saturation in TFIs gives us the features of both effects. Characteristic 1–2 ps decay of the shift confirms the metal clusters nature of the effect [23, 24]. Maximal NLO effects in nanocrystals are increased with size reduction, and are enhanced by extra
excitation power, pulse durability [20, 22] and γ-irradiation [19]. These effects can be explained by accumulation of colloids and electrons at nanocrystal boundaries.

Figure 8. NLO characteristics detected for: (a) reference films (1), multilayers (2, 3), (b) multipeak TFIs with c-oriented cubic CdS nanocrystals of defined sizes. (c) Transmission spectra of the TFI before (B) and at time of excitation (time delay between the excitation and registration is zero) (A).

Impurities and nonstoichiometric excess tend to be expelled from the small crystalline core, as the distance a defect or impurity must move to reach the surface of a nanocrystal is very small. Self-purification of the nanocrystals can be explained through technological arguments and is an intrinsic property of defects in nanocrystals, prepared by the methods of alternative deposition of nanolayers with thicknesses 150–10 nm. Post-growth thermal treatments, ionising radiation bombardment and light irradiation of the nanocrystals are the next tools for the nanocrystal purification, metal colloids formation and the nanocrystal properties modification.

5. Conclusion
The c-axis highly oriented thin-film multilayer structures composed of dielectric (LiF, CaF$_2$) and semiconductor (CdS) nanocrystals prepared by thermal evaporation show enhanced photo-response, which is related to high-quality nanocrystals, but at the same time depends strongly on the defect content, especially as far as their 1–2 ps nonlinearities are concerned. Critical factors are nanocrystal purity as well as metal impurities and nonstoichiometric excess collection in their boundaries regions. The nanostructures (more high-quality than original bulk structures) can be part of efficient emitting elements, detectors and modulators of light, as well as a novel type of dosimeters. Such optically stimulated luminescence dosimeters (integrable with SiO$_2$:OH fibres) provide capability for remote monitoring radiation locations which are difficult to access and hazardous. These devices are relatively simple, small in size, cheap in production and have low power consumption. Moreover, they are also suitable for space radiation dose exploration. In addition, they can be used in other radiation measurements, and have interesting perspectives in the case of periodical nanostructures composed of the nanolayers of CdS and LiF nanocrystals with high-contrast indices of refraction (figure 1c).

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