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The Glow Discharge Application for Formation of a Thin Luminescent Layer in Wide Gap Crystals

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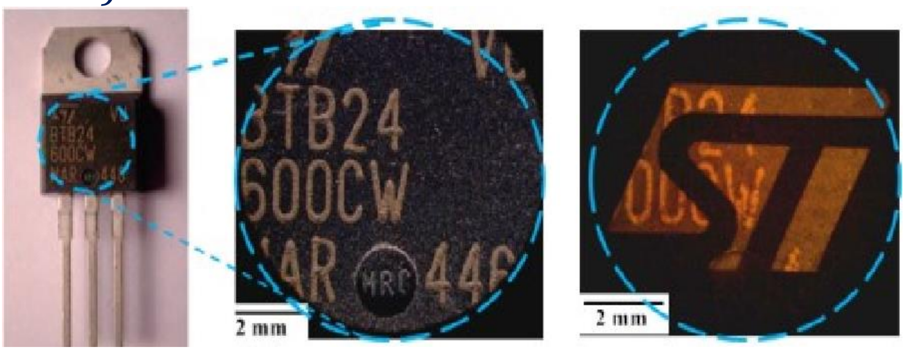
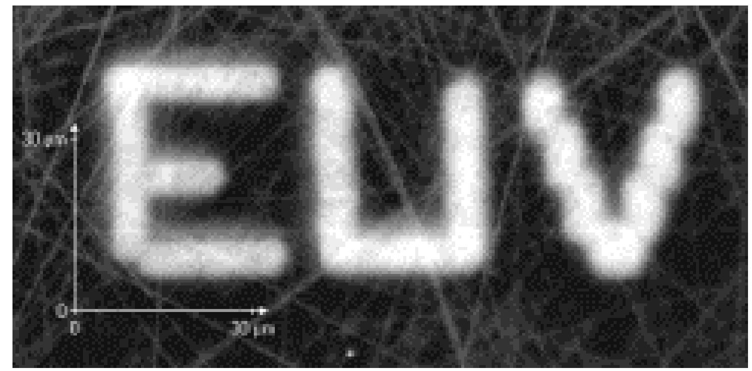
Introduction

Invisible marking system by extreme ultraviolet radiation: The new frontier for anti-counterfeiting tags. /*P. Di Lazzaro, S. Bollanti, F. Flora ... (Journal of Instrumentation. **2016**)*



Left: transistor covered by a optically transparent and patterned LiF film.
Middle: detail of the transistor seen by an optical microscope 2,5X.
Right: the same area of the middle figure, as seen at fluorescence microscope that allows to see the latent patterned logo ST.

Formation and direct writing of color centers in LiF using a laser-induced extreme ultraviolet plasma in combination with a Schwarzschild objective. / *F. Barkusky , C. Peth, K. Mann (Rev. Sci. Instrum.. **2005**)*



Direct writing of color centers in LiF, visualized with a laser scanning microscope. The EUV spot has a diameter of 5 mkm

Formation of color centers in a thin layer of LiF crystals under VUV radiation from a barrier discharge/ *E.V. Milyutina, A.F. Petrovskii, A.L. Rakevich, E.F. Martynovich, Technical Physics Letters, **2014**)*

Formation of a Thin Luminescent Layer in LiF Crystals under Glow Discharge Radiation/ *A. A. Tyutrin, D. S. Glazunov, A. L. Rakevich, E. F. Martynovich, Technical Physics Letters. **2018**.)*

The Purpose and Tasks

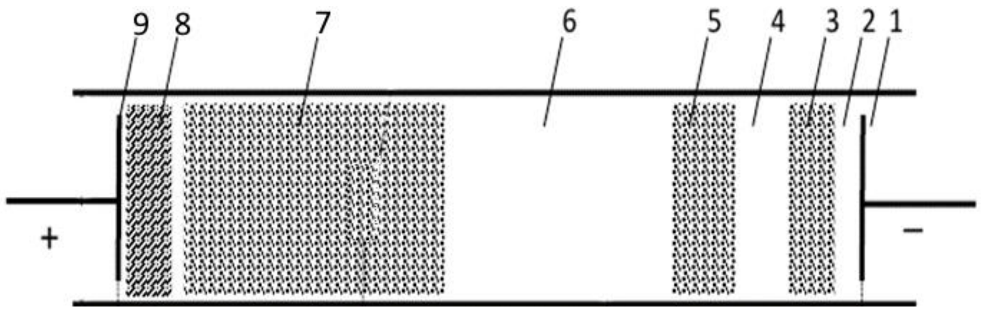
The purpose of the work was:

- to investigate the glow discharge application for formation of a thin luminescent layer in wide gap crystals.

The tasks were:

- to study the axial intensity distribution of the VUV radiation of a glow discharge in air, depending on the voltage and pressure.
- to identify glow discharge zones in which the formation of defects is most efficient.
- to determine the types of formed color centers;
- to study the spectral and kinetic characteristics of their luminescence;

The Glow Discharge and its Features



An electric glow discharge tube featuring its most important characteristics:

(1) An anode and cathode at each end,
(2) Aston dark space,
(3) Cathode glow,
(4) Cathode dark space,
(5) Negative glow,
(6) Faraday space,
(7) Positive column,
(8) Anode glow,
(9) Anode dark space.

- **The negative glow** contains the lines of the molecular nitrogen ions N_2+ (the so-called first negative system) **391 nm**, as well as the line **357 nm**, belonging to the second positive system of the neutral nitrogen molecule N_2. **+(427, 470 nm)**
- **The Faraday dark space** contains the lines of the second positive nitrogen system at **399 nm** and **326 nm**.
- **In the positive column**, the brightest lines are the lines of the second positive system **357 nm., 380 nm., 399.**, as well as the lines of the first positive system.**+ 662 nm**.

Uac = 1,25 kV
Iac = 1мA
P= 530 Pa

Image of the glow discharge

The Glow Discharge and its Features

Interband ionization

Electrons and ions
(E > Eg ~14 eV)

Photons
(E > E_g ~14 eV)

The main mechanism of defect formation in alkali-halide crystals is the decay of anionic excitons into Frenkel pairs, which is accompanied by the further processes of recharging, migration and aggregation with the formation of stable luminescent aggregate color centers.

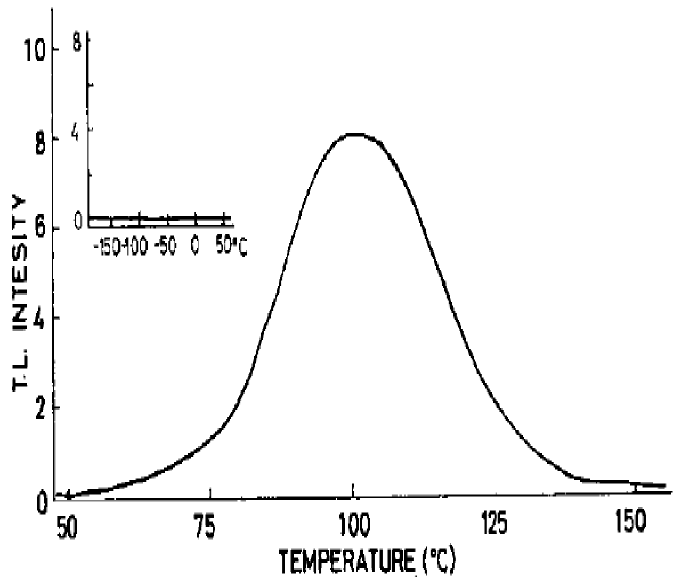
For this reason, the formation of defects requires **interband ionization**, which can be potentially provided by three glow discharge components: **electrons, ions**, and **photons** with an energy exceeding the bandgap width, which is 13.6 or 14.2 eV for LiF according to different data.

Formation of a Thin Luminescent Layer in LiF Crystals under Glow Discharge Radiation / A. A. Tyutrin, D. S. *Glazunov, A. L. Rakevich, E. F. Martynovich, Technical Physics Letters.* ***2018****.)*

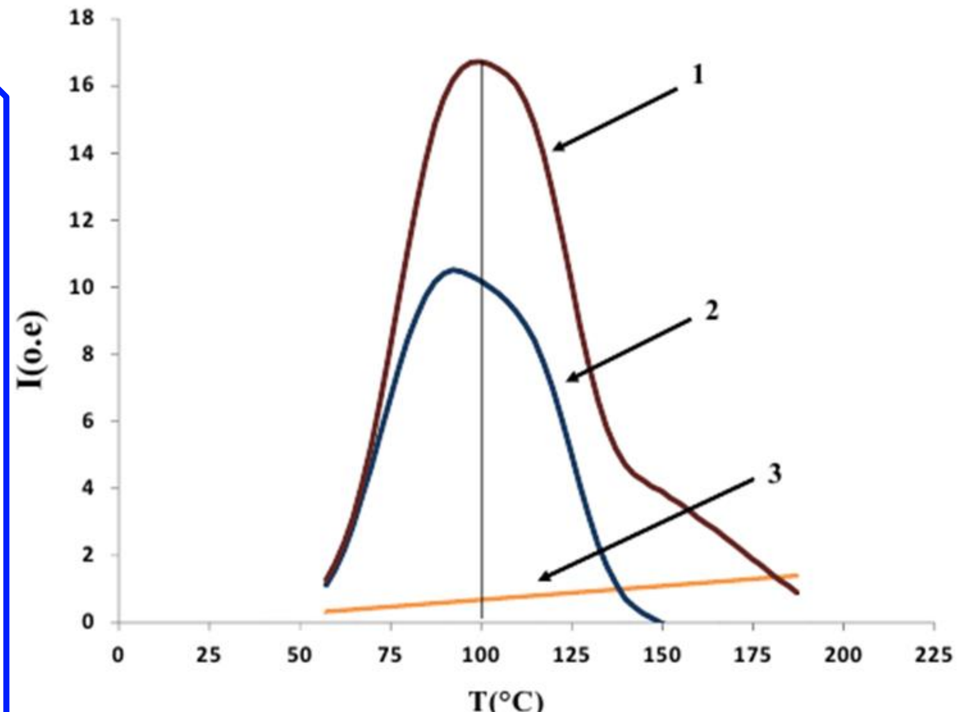
The Axial VUV Radiation Intensity Distribution of a Glow Discharge

How do we plan to obtain VUV radiation distribution?

$CaSO_4 \cdot Mn$ thermoluminophor is sensitive to radiation in the spectral region of shorter than **130 nm.**
Maximum sensitivity to : λ = **103 nm**.
Peak of thermoluminescence: **λ = 500 nm.**



Glow curves for $CaSO_4$ (Mn) - Thermoluminescence
(Nakajima T. // J. Nucl. Sci. Technol. 1968. V. 5. N 7. P. 360–364)

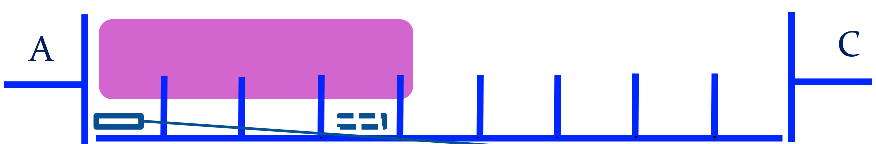


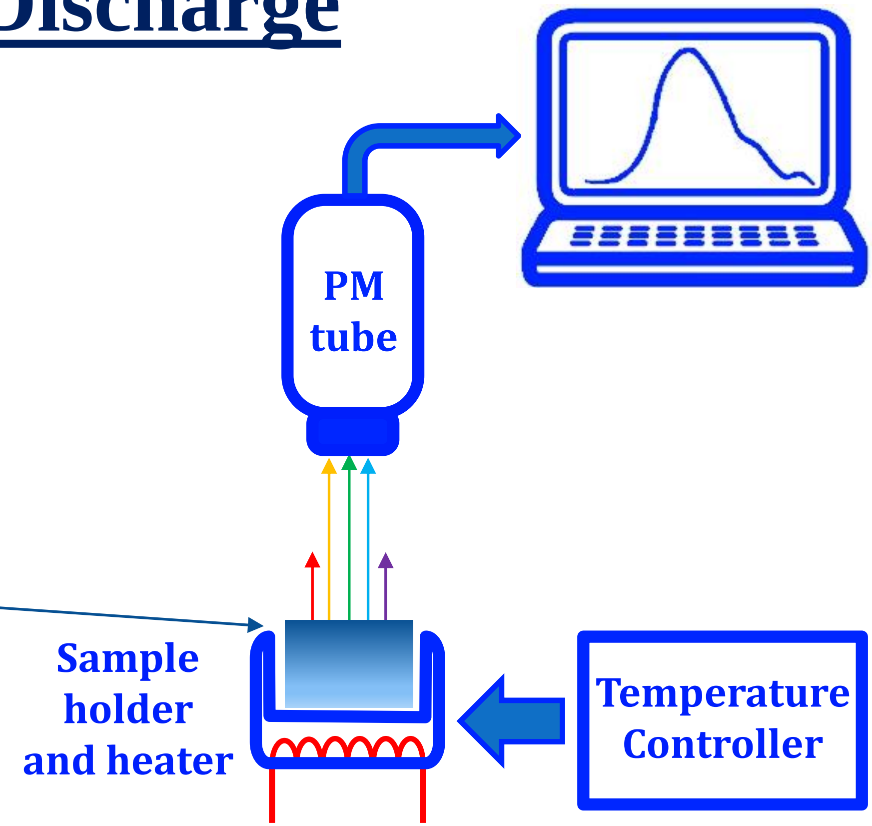
Glow curves for $CaSO_4$: Mn thermoluminescence after irradiation by :

- glow discharge at **1** - 2.8 kV, **2** - 1.6 kV,
- UV lamp at wavelength **200 nm – 3.**

The Axial VUV Radiation Intensity Distribution of a Glow Discharge

A copper plate coated with a thermoluminophor layer was placed in a miniature diaphragm cell. The cell was installed on the internal wall of the discharge tube at different distances between the electrodes.

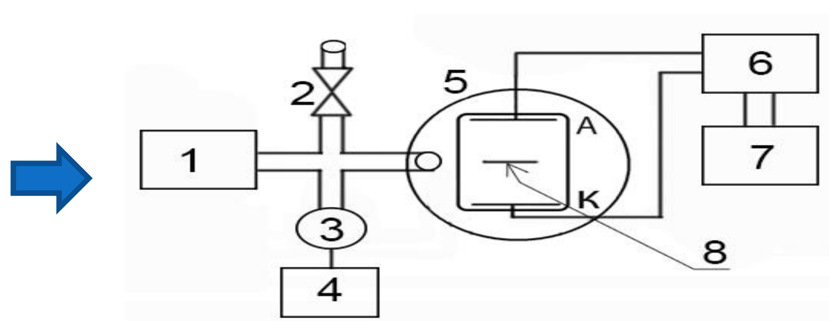




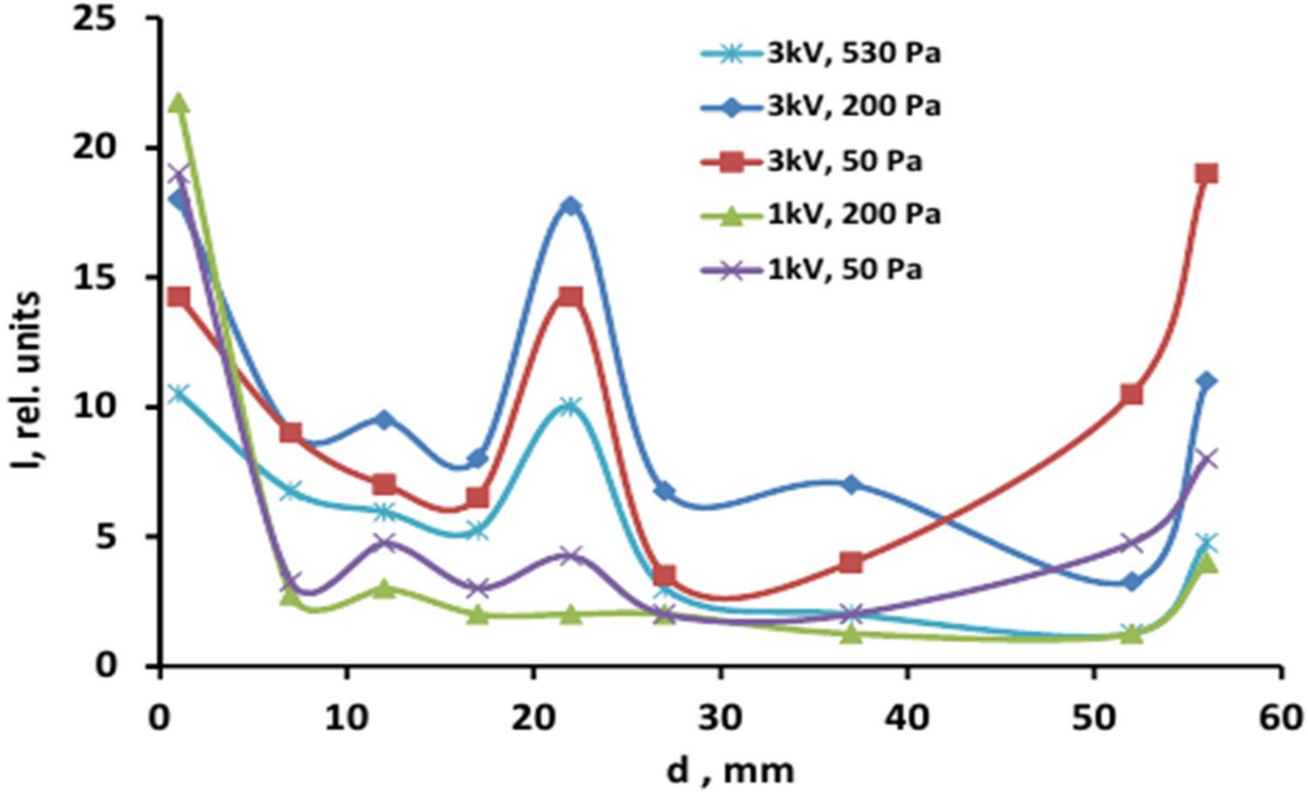
Thermoluminescence measurement scheme

The design of this cell provided the detection of radiation emitted predominantly at a right angle to the discharge direction.

Block diagram of the experimental setup: **(1)** Vacuum pump, **(2)** Valve, **(3)** Vacuum gauge, **(4)** Thermal conductivity gauge, **(5)** Vacuum chamber, **(6)** High-voltage source, **(7)** block of the measurements, **(8)** sample.



The Axial VUV Radiation Intensity Distribution of a Glow Discharge

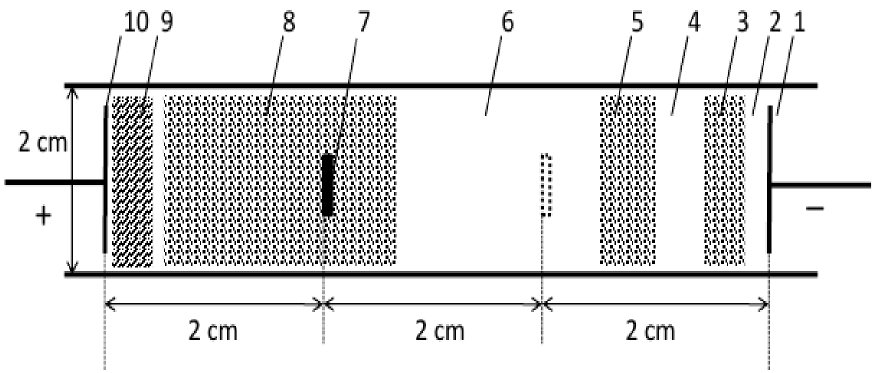


U = 1 - 3 kV
P = 50 – 530 Pa

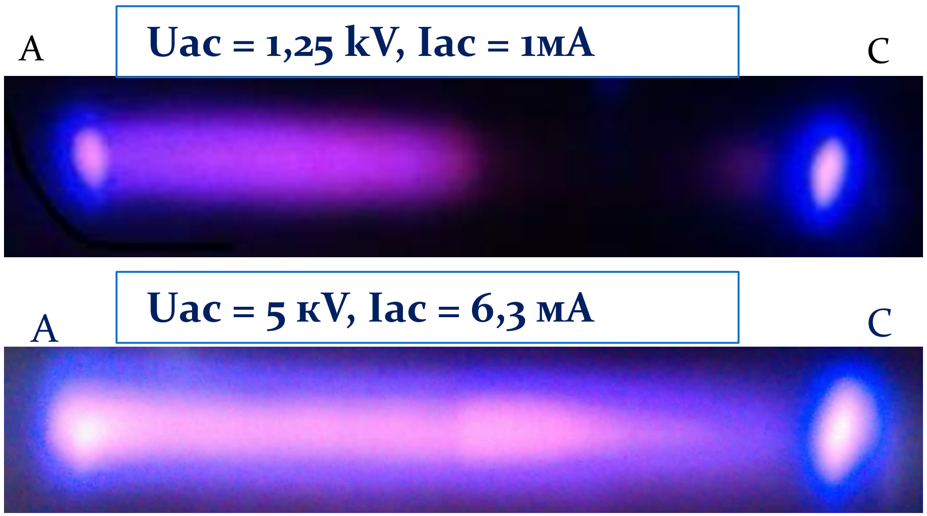
VUV radiation intensity distribution along a discharge by $CaSO_4 \cdot Mn$ thermoluminophor at different voltages and pressures in the experiment (left is the anode, and right is the cathode)

- The curves of the intensity distribution of the VUV radiation at pressures of 50 - 530 Pa do not differ much from each other in the region of the positive column and in the Faraday dark space, but they begin to noticeably differ in the cathode region. It can be seen that the intensity of the VUV radiation increases with decreasing pressure in the cathode region of a glow discharge.
- The intensity of the VUV radiation also increases with increasing voltage on the electrodes.

Formation of a Luminescent Layer in LiF Crystals by The Glow Discharge

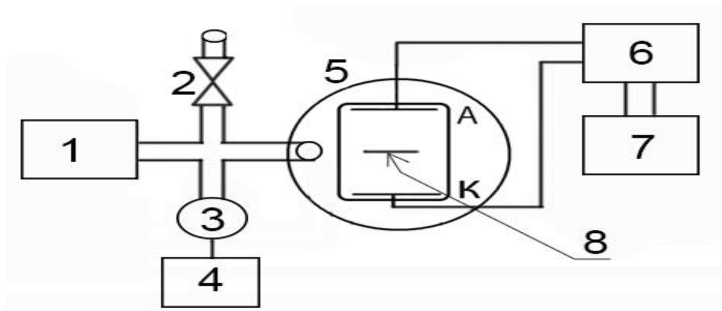


An electric glow discharge tube featuring its most important characteristics: **(1)** An anode and cathode at each end, **(2)** Aston dark space, **(3)** Cathode glow, **(4)** Cathode dark space, **(5)** Negative glow, **(6)** Faraday space, **(7)** specimen, **(8)** Positive column, **(9)** Anode glow, **(10)** Anode dark space.



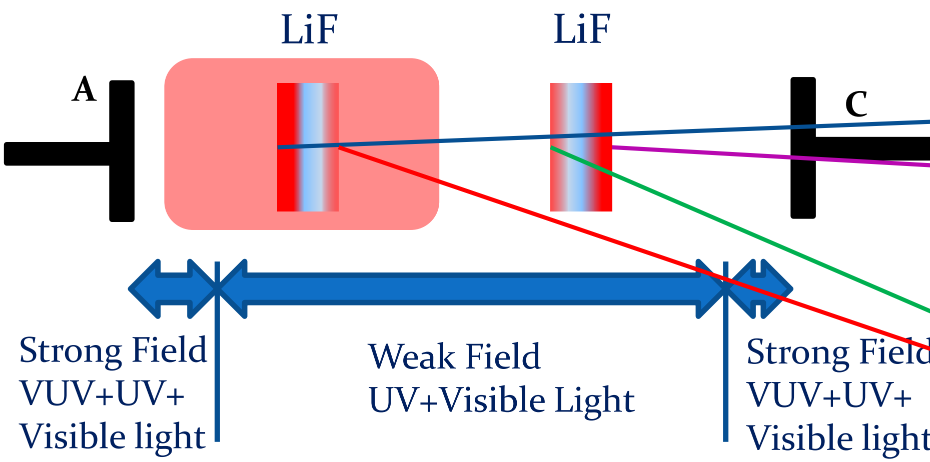
Images of the glow discharge at different voltages in the experiment

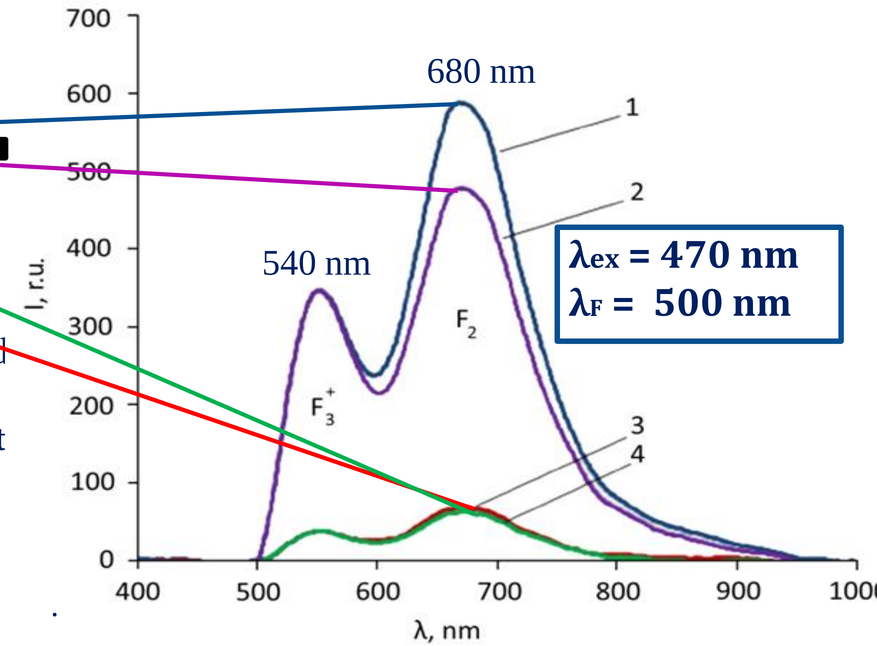
Block diagram of the experimental setup: **(1)** Vacuum pump, **(2)** Valve, **(3)** Vacuum gauge, **(4)** Thermal conductivity gauge, **(5)** Vacuum chamber, **(6)** High-voltage source, **(7)** block of the measurements, **(8)** specimen.



Formation of a Luminescent Layer in LiF Crystals by The Glow Discharge

Luminescence spectra and kinetics of irradiated LiF crystals





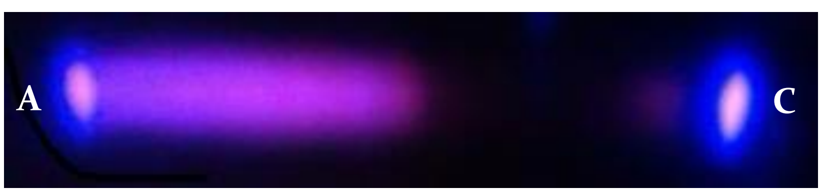
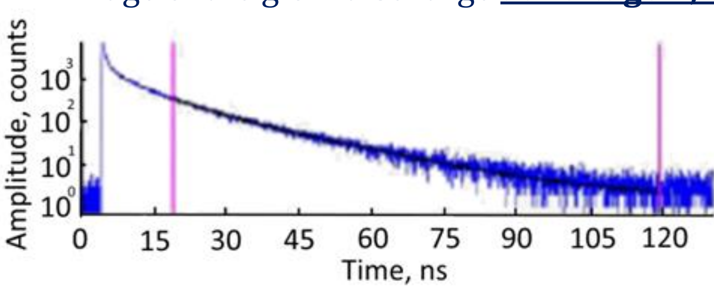


Image of the glow discharge **at voltage 1,25 kV**



Ampl.1	136
Lifet.1	6.8
Ampl.2	201
Lifet.2	17.8
Backgr.	1.58

Kinetics of the color centers luminescence at picosecond laser exitation.

Excitation with picosecond laser pulses at a wavelength of 470 nm, light filter with a transmittance from 500 nm.

(1) and (3) - for a specimen in the positive column,

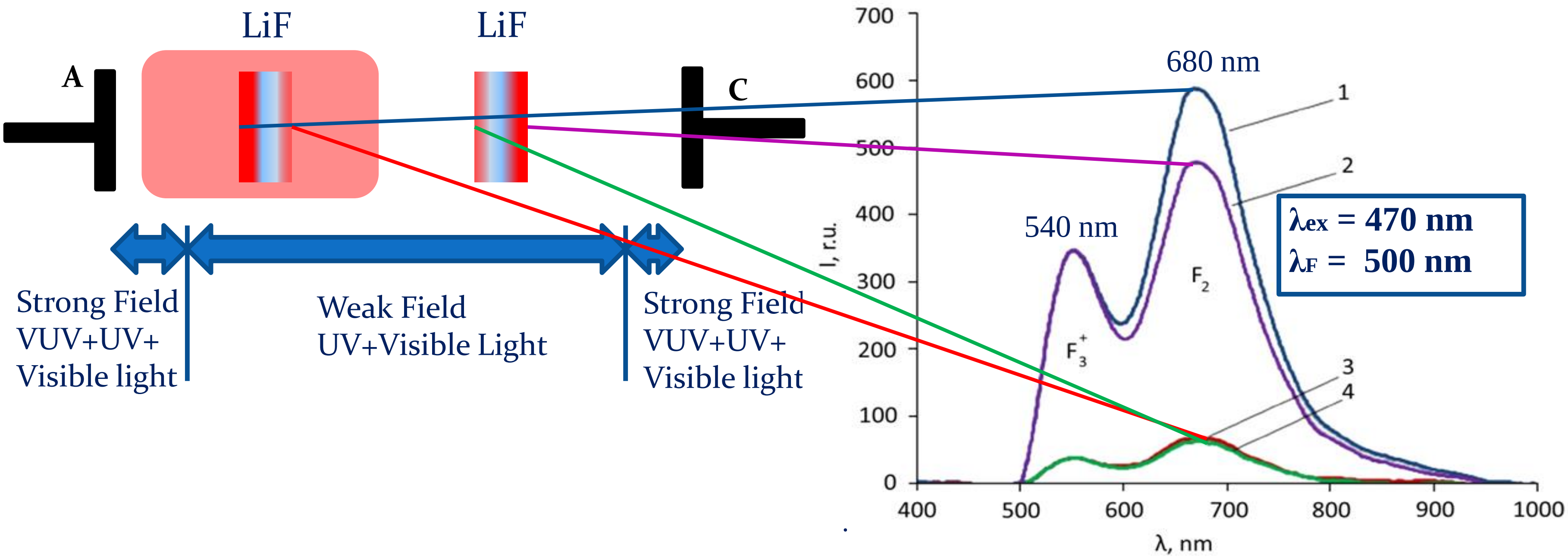
(2) and (4) - in the Faraday dark space,

(1) and (4) - for the anode side (turned towards the anode) and

(2) and (3) - for the cathode side.

Formation of a Luminescent Layer in LiF Crystals by The Glow Discharge

Luminescence spectra and kinetics of irradiated LiF crystals



- ❖ The LiF absorption bandgap contains series of nitrogen and oxygen radiation lines at **99.1–64.4** and **98.8–78.7** nm, respectively.
- ❖ Electron–hole pairs and excitons formed upon the absorption of radiation in these lines form anionic Frenkel defects, which are an initial product for the creation of aggregate color centers.
- ❖ This is explained by that the near-electrode regions are characterized by the strong electric field, which enables the electrons to acquire the energy sufficient for the excitation of higher energy levels of nitrogen and oxygen.

Conclusions

- It was obtained the axial VUV radiation intensity distribution of a glow discharge with a wavelength shorter than 130 nm using the thermally stimulated luminescence method.

- **The VUV radiation intensity distribution has maximum values in the near-electrode regions of the discharge in the pressure range of 50–530 Pa and voltages of 1–3 kV. The stratified positive column of glow discharge is also a region of intense VUV radiation at high voltage. Therefore, the coloring of crystals near these regions is most effective.**

- It has been established that basically two types of color centers are formed on the surface layers of crystals: F_2 and F_3^+ color centers with luminescence bands peaking at ~ 680 and 530 nm, respectively.

- **It is shown the color centers are mainly formed by photon-induced mechanism, namely, defect formation under the action of vacuum ultraviolet (VUV) radiation.**

- Glow discharge can be successfully used to form thin luminescent layers on the surface of transparent insulators for various scientific and practical applications.

Thanks for your attention!