TAMPERE UNIVERSITY OF TECHNOLOGY

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Irradiation with alpha particles of Yb^{3+} and Er^{3+} co-doped phosphate glasses *Preliminary results*

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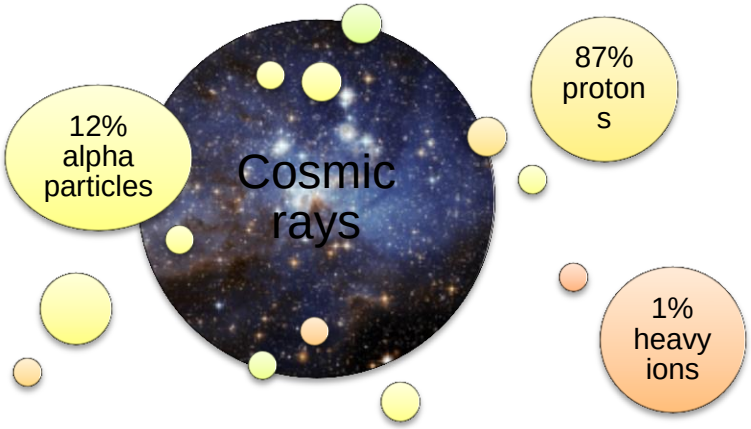
Motivation

The research group at the National Institute for Laser, Plasma and Radiation Physics is running for several years tests of ionizing radiation effects on optoelectronic materials components and devices.

Under the umbrella of the COST Action MP1401 the two teams from Finland and Romania perform investigations on irradiated active glasses for possible use in space missions.

Glasses (melted in Finland) irradiated (in Romania) using alpha particles.

Irradiation impact investigations on:
- surface quality,
- optical and THz absorption;
- luminescence
- stability of the radiation induced effects in time and after heating;



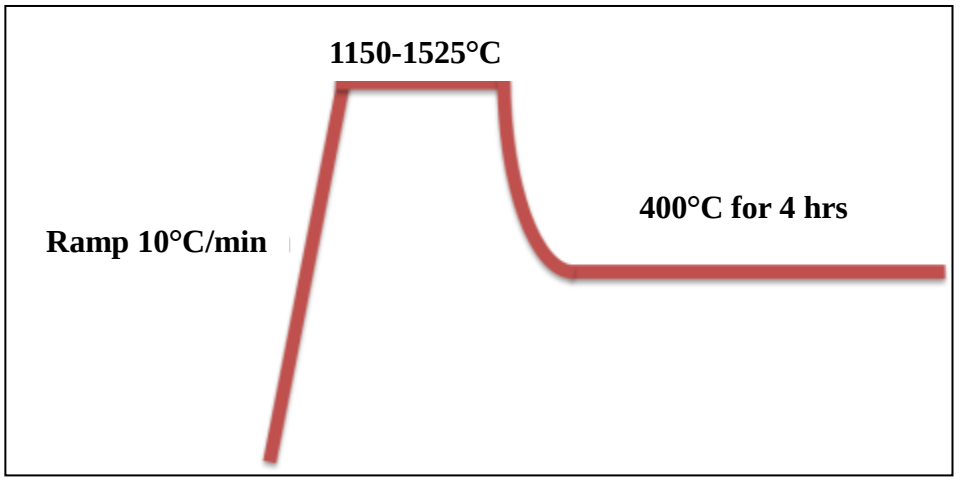
Glass Melting

Glass composition in mole %

$(98-x)(0.50P_2O_5\text{-}0.40SrO\text{-}0.10Na_2O)\text{-}0.5Er_2O_3\text{-}1.5Yb_2O_3\text{-}xZnO$

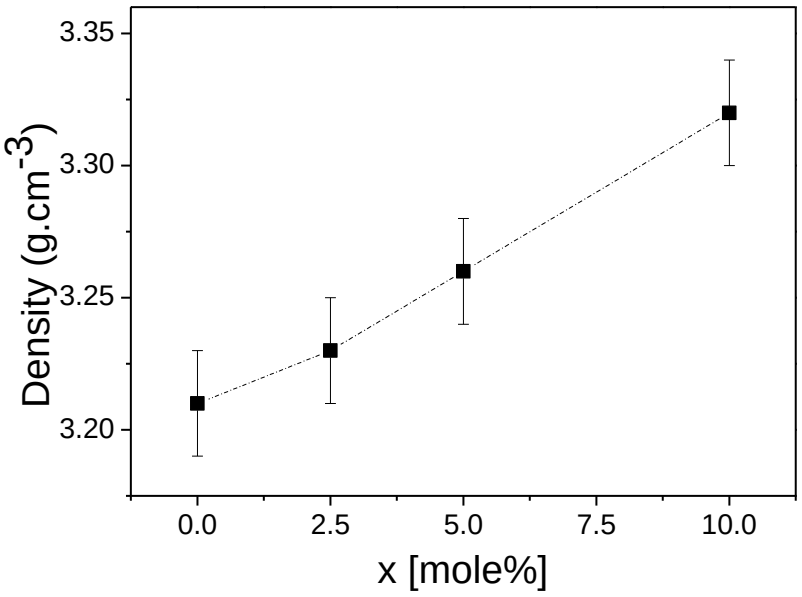
$Sr(PO_3)_2$ $NaPO_3$ ZnO

Er_2O_3 Yb_2O_3

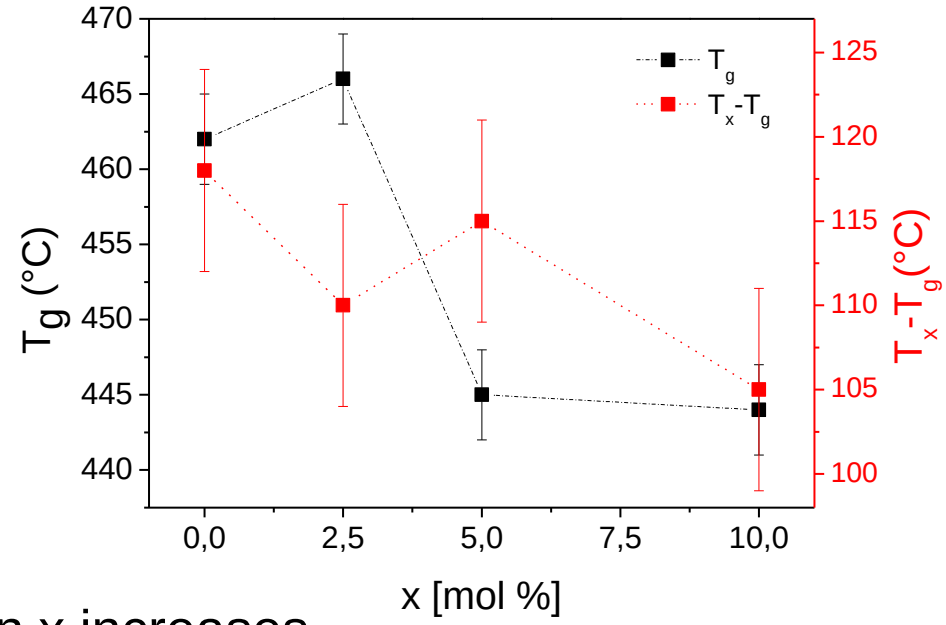


Physical and thermal properties

Density



Thermal properties



When x increases

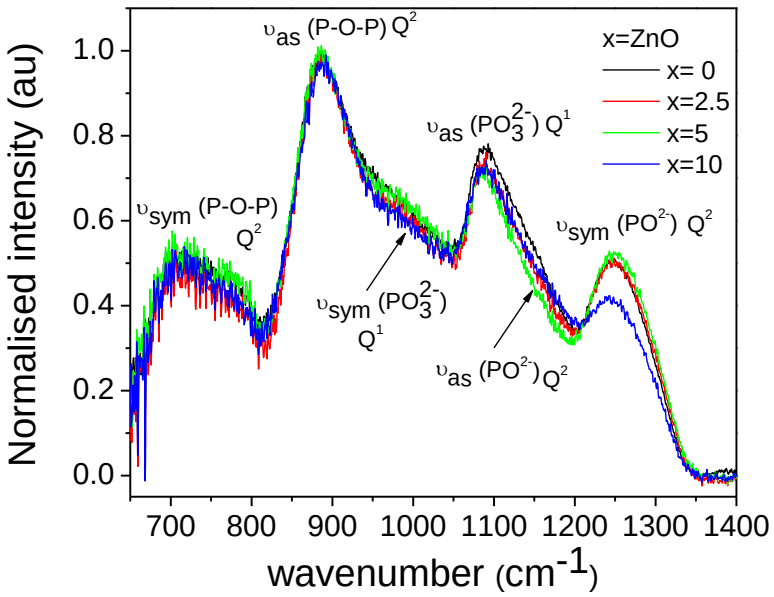
Density increases due to partial replacement of P_2O_5, Na_2O and SrO in glass network by the heavier ZnO

T_g decreases
$\Delta T = T_x - T_g > 100°C$
→ good stability against crystallization

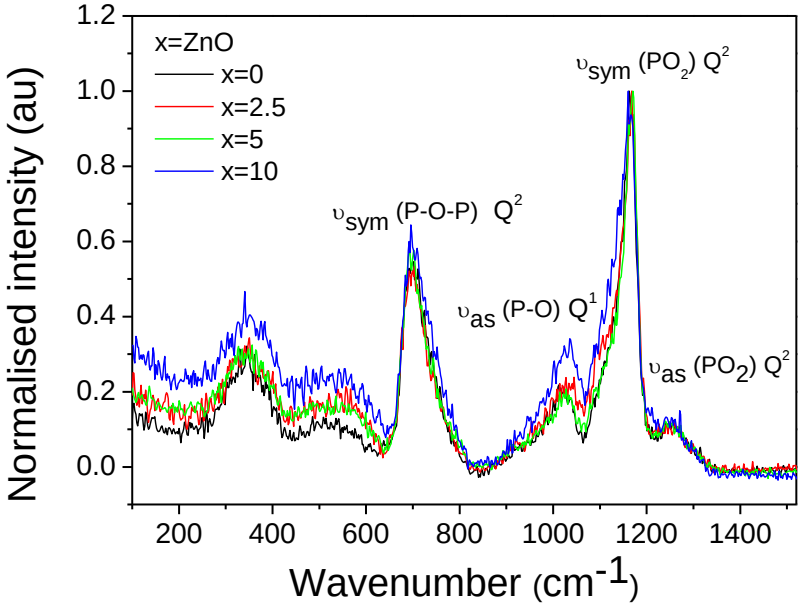
Glass system: $(98\text{-}x)(0.50P_2O_5\text{-}0.40SrO\text{-}0.10Na_2O)\text{-}0.5Er_2O_3\text{-}1.5Yb_2O_3\text{-}xZnO$

Structural Properties

IR spectra



Raman spectra



When x increases

→ decrease in intensity of the bands at 1085 and 1250cm^{-1}

When x increases

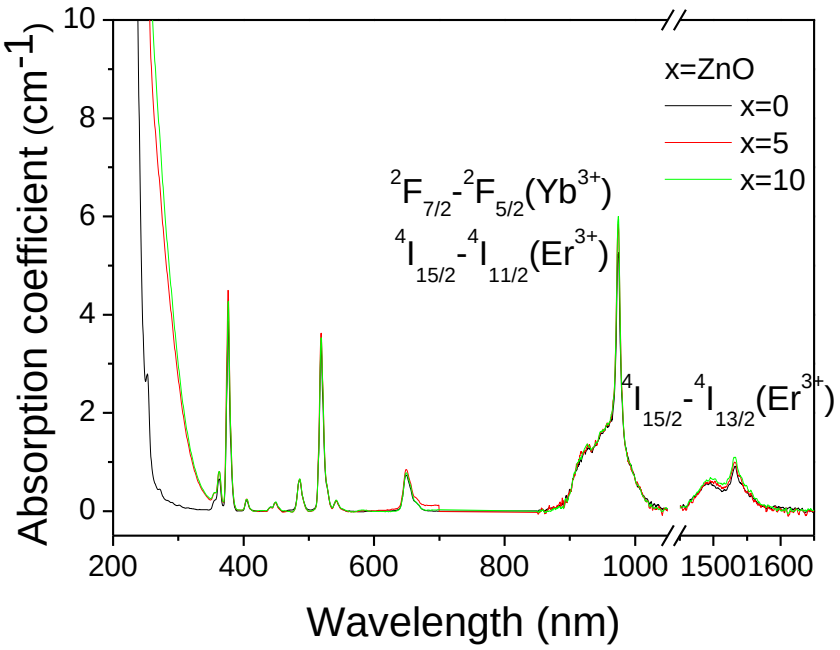
→ increase in intensity of all the bands

Metaphosphate structure (Q^2 and Q^1 units), ZnO acts as a network modifier in agreement with the decrease in T_g

Glass system: (98-x-y)(0.50P_2O_5-0.40SrO-0.10Na_2O)-0.5Er_2O_3-1.5Yb_2O_3-xZnO-yY_2O_3

Optical Properties

Absorption spectra



Band gap shifts to longer wavelength with an increase in x due to the depolymerization of the phosphate network

Absorption cross-section

$$\sigma_{abs}(\lambda)=\alpha_{abs}(\lambda)/N$$

Absorption cross-section

Absorption coefficient

Number of absorbing ions

When x increases

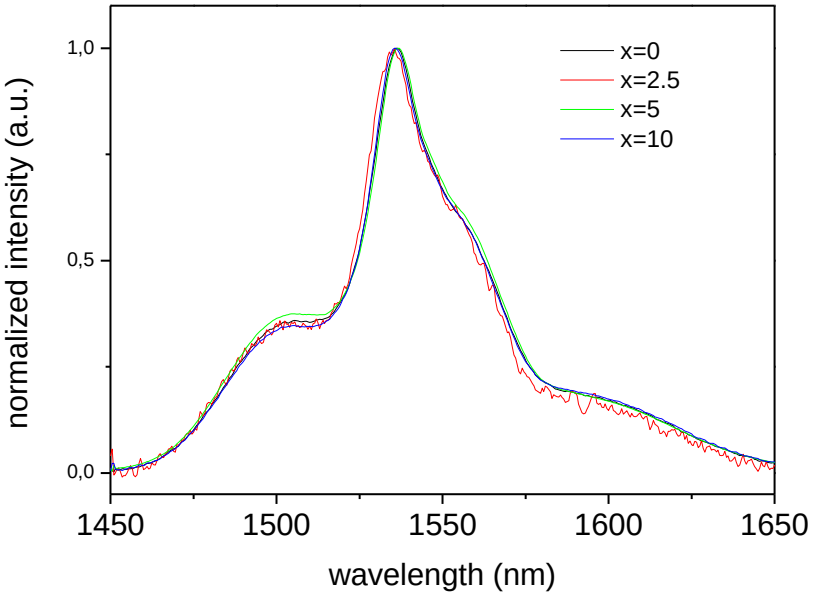
→ no variation in absorption cross-section within accuracy of measurement

- σ_{abs} (980 nm)~8.8 ×10^{-21} cm^2;
- σ_{abs} (1.5 μm) ~6.6 × 10^{-21} cm^2

Glass system: (98-x)(0.50P_2O_5-0.40SrO-0.10Na_2O)-0.5Er_2O_3-1.5Yb_2O_3-xZnO

Spectroscopic Properties

Normalized emission spectra
(λ_{exc}=980nm)



x	lifetime of Er^{3+}:$^4I_{13/2}$[ms] (±0.2ms)
0	3,47
2,5	3,19
5	2,98
10	3,27

No change in the shape of the emission band and in the lifetime of Er^{3+}:$^4I_{13/2}$

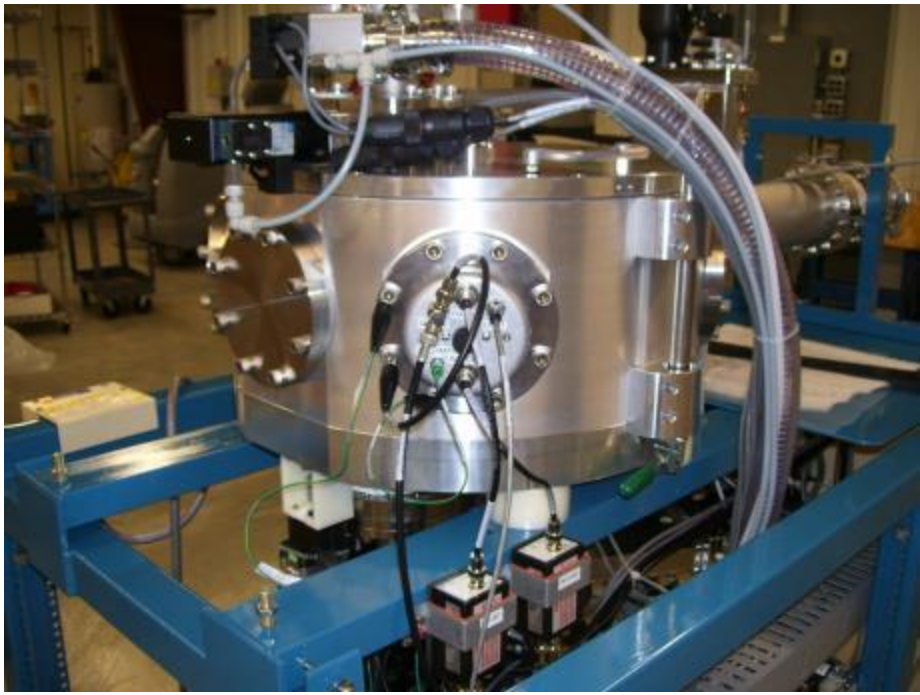
→ "Er^{3+} and Yb^{3+} ions' sites are not strongly affected by the change in the glass composition"

Glass system: (98-x)(0.50P_2O_5-0.40SrO-0.10Na_2O)-0.5Er_2O_3-1.5Yb_2O_3-xZnO

Irradiation set up

Irradiation by alpha particles

- 3 MeV U-120 Cyclotron accelerator,
- beam current of 3 nA
- the total fluence reached during this study was 10^{12} α/cm^2.



U-120 Cyclotron accelerator

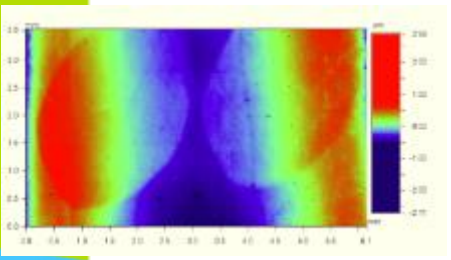
Alpha particles source *Sample holder*

Inside of vacuum chamber

Date of irradiation: May 5th, 2017

Surface profile measured on June 28th 2017

Surface profile of the glass with x=1.25 (Zn1.25) measured using Veeco using stich option



6mm

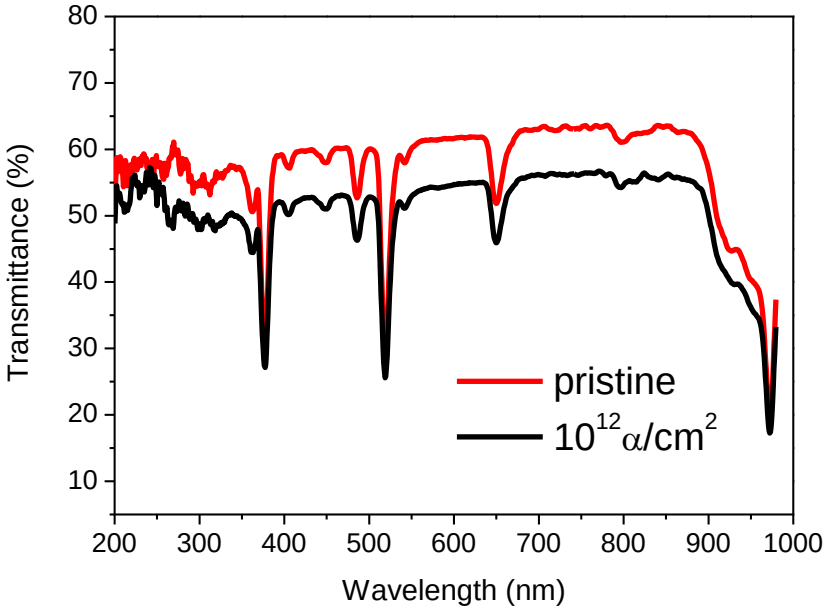
Surface expansion ~185-250nm

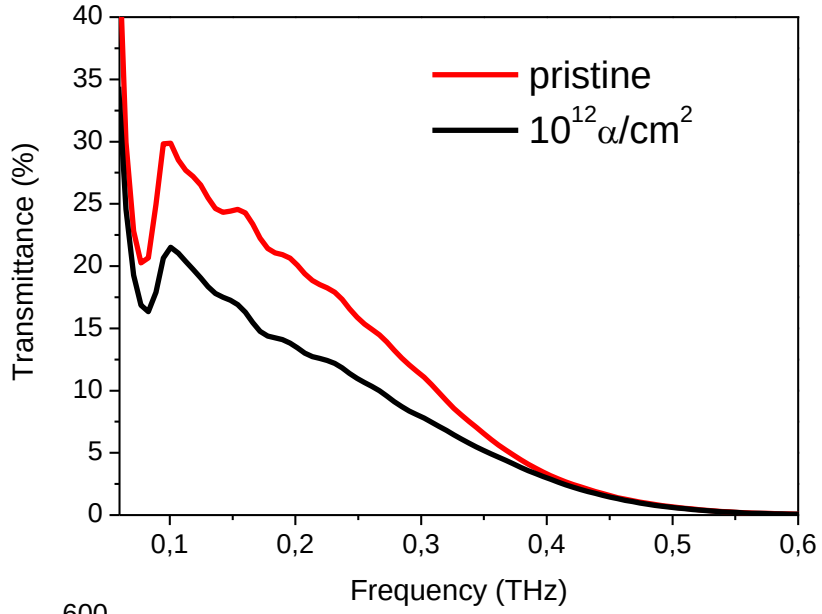
Ra: arithmetical mean deviation of the assessed profil
Rq: root mean squared
Rz: average distance between the highest peak and lowest valley
Rt: Maximum Height of the Profile

Irradiation leads to increase in roughness

	Ra (nm)	Rq (nm)	Rz (nm)	Rt (nm)
Non-irradiated	38	46	431	505
Irradiated spot 1	244	276	1130	1440
Irradiated spot 2	219	250	1470	2640

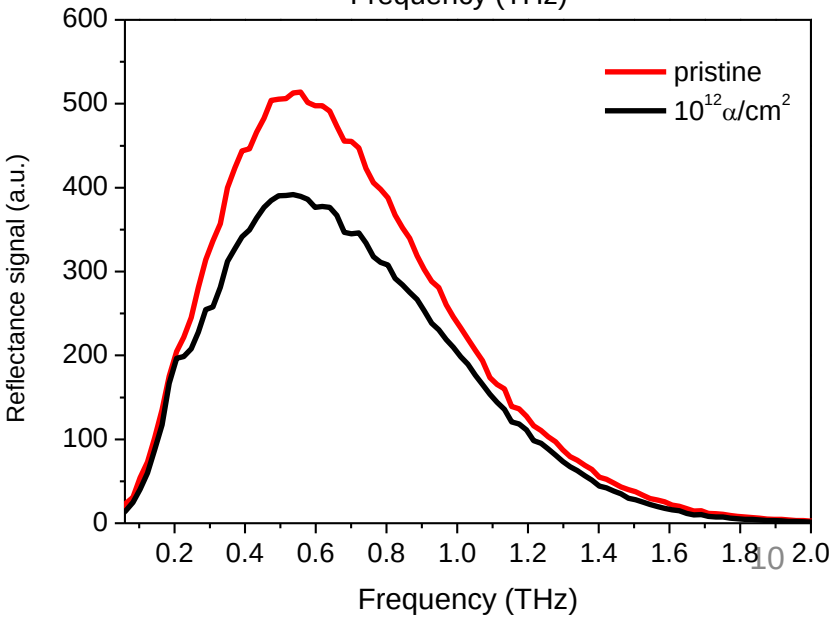
Optical and THz properties of Zn1.25





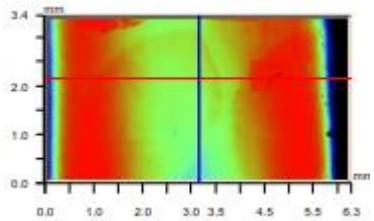
A decrease of around 10% was noticed after irradiation on the transmittance results for both ranges THz and UV-NIR.

Also the reflectance in THz range decreased after irradiation.

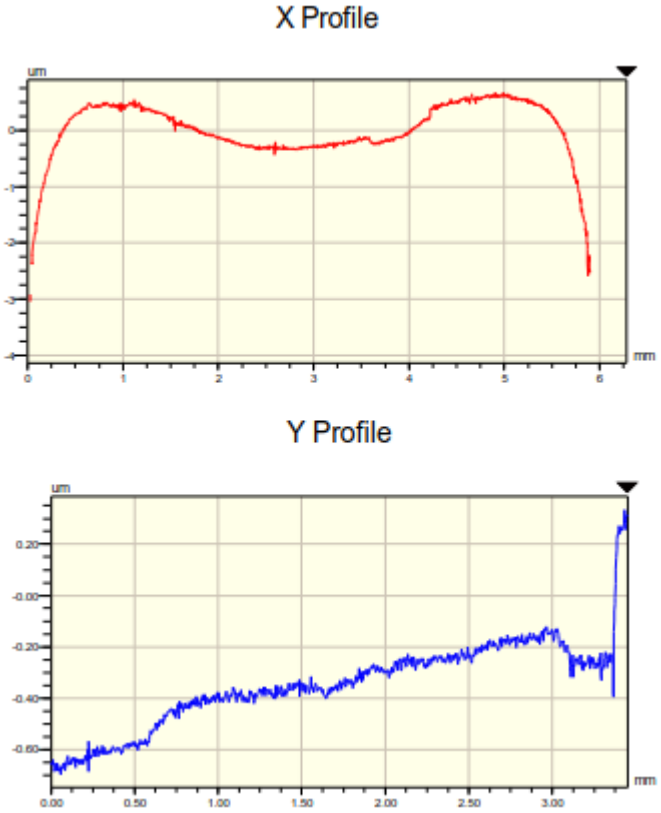


Surface profile measured on June 28th 2017

Surface profile of the glass with x=2.5 (Zn2.5) measured using Veeco using stich option



6mm



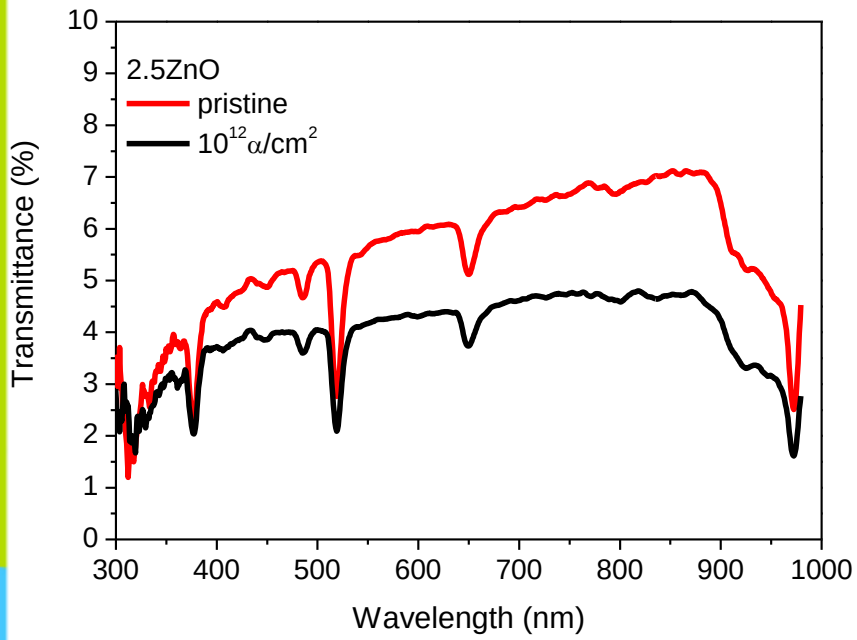
Surface expansion ~150-200nm (slightly less than in Zn1.25)

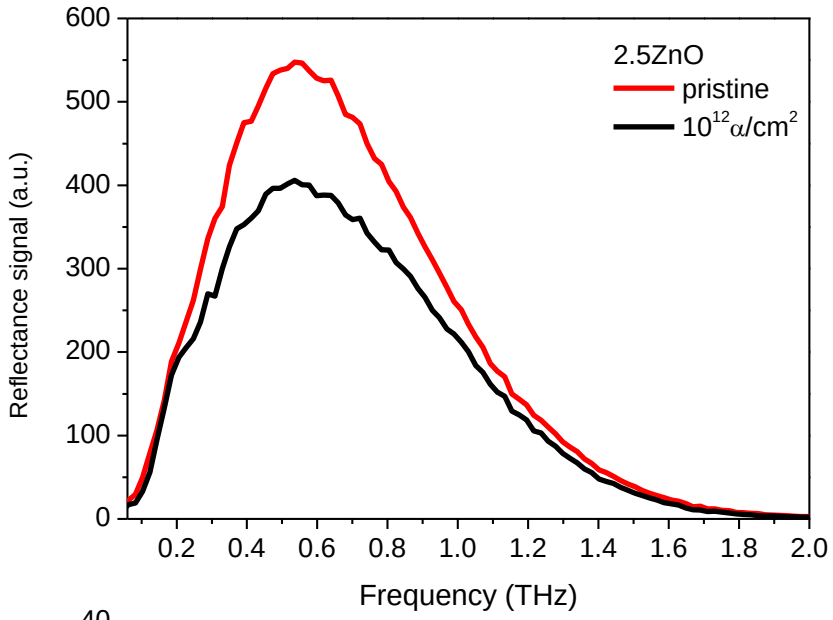
Ra: arithmetical mean deviation of the assessed profil
Rq: root mean squared
Rz: average distance between the highest peak and lowest valley
Rt: Maximum Height of the Profile

Irradiation leads to an increase in roughness (Ra and Rq) (similar roughness than in irradiated Zn1.25)

	Ra (nm)	Rq (nm)	Rz (nm)	Rt (nm)
Non-irradiated	83	96	478	609
Irradiated spot	228	266	1860	2880

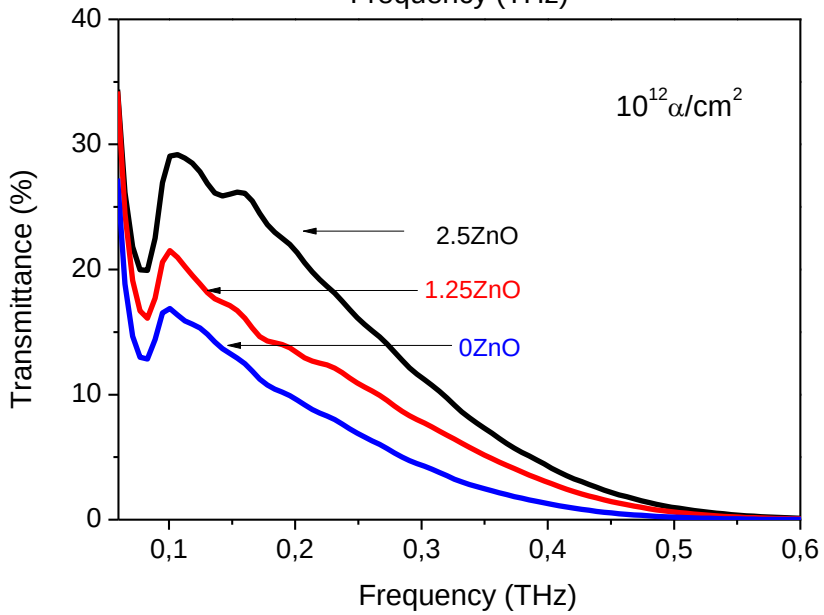
Optical and THz properties





The transmittance decreased after irradiation in UV-NIR range. Also the reflectance in THz range decreased after irradiation.

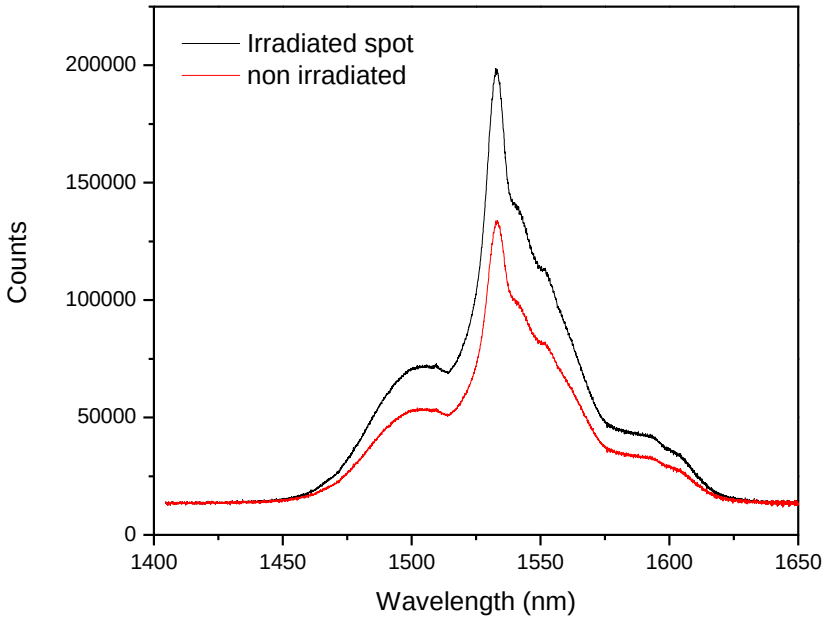
In THz range the transmittance for irradiated samples increased when the ZnO concentration increased.



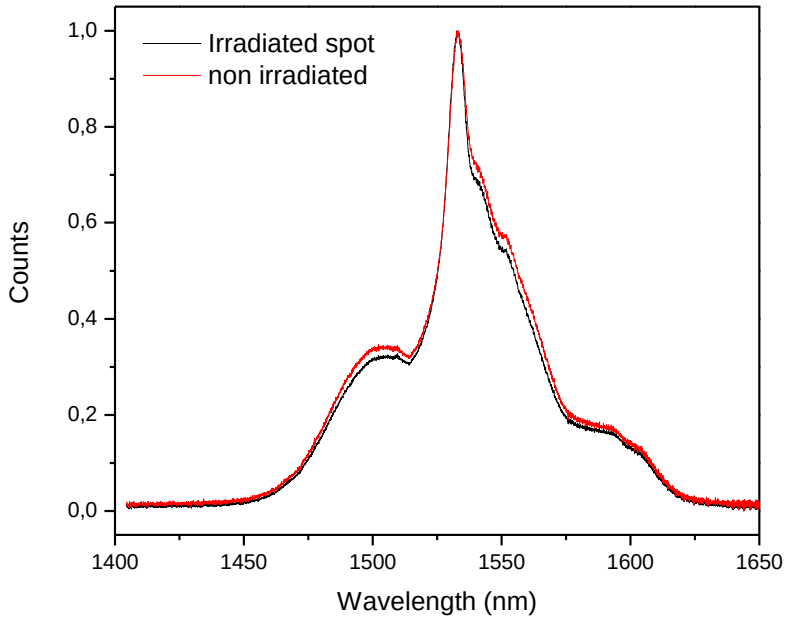
Microluminescence (measured on Aug 9th 2017)

Excitation: 72mW at 532nm; 40x objective, spot size ~500-1000nm

Emission spectra of Zn2.5 glass



Normalized emission spectra of Zn2.5 glass



Different intensity and shape of emission measured in the irradiated and non-irradiated areas only in the Zn2.5 glass (TBC with new experiments!)

Surface profile measured on August 10th 2017

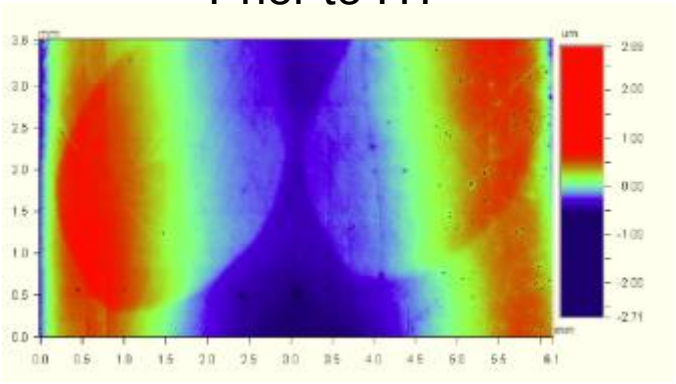
Samples stored in ambient atmosphere at room temperature in dark

Irradiated					
	Ra (nm)	Rq (nm)	Rz (nm)	Rt (nm)	Max Expansion (nm)
Zn1.25-June	219	250	1470	2640	~180-250
Zn1.25- August	121	154	621	642	~180
Zn2.5-June	228	266	1860	2880	~150-200
Zn2.5-August	150	192	949	990	~150-180

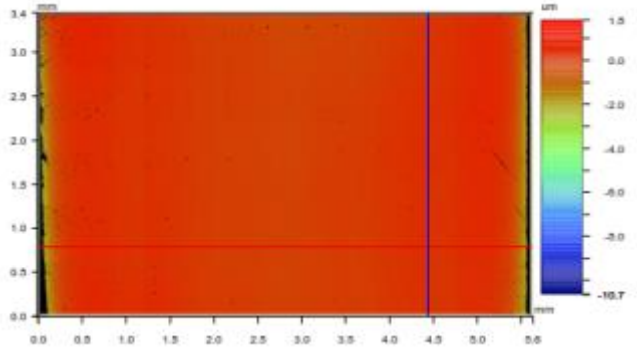
Small decrease in the surface photo-expansion and roughness overtime

Surface profile after annealing at 400°C for 8h

Prior to HT



After HT



Zn1.25 taken as an example

Zn1.25	Ra (nm)	Rq (nm)	Rz (nm)	Rt (nm)
Prior to heat treatment				
non-irradiated area	38	46	431	505
irradiated spot	121	154	621	642
After heat treatment				
non-irradiated area	**32**	**37**	**181**	**192**
former irradiated spot	**124**	**138**	**577**	**720**

After HT

- Spots are not visible anymore
- No change in the surface roughness

Same observed in Zn2.5 glass

Conclusion

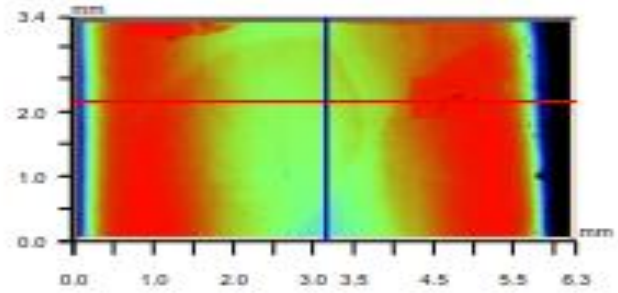
New phosphate glasses were prepared, characterized and irradiated using alpha particles

- Addition of ZnO leads to the depolymerization of the network with a decrease of T_g but has no impact on the rare-earth sites
- Alpha irradiation leads to
  - Surface expansion and an increase in surface roughness (no impact of the ZnO content (1.25 vs 2.5mol%) on glass photo-response)
  - Decrease in transmission in visible and THz ranges of about 10%.
  - Changes in the Er^{3+} emission properties (shape and intensity) (to be confirmed)

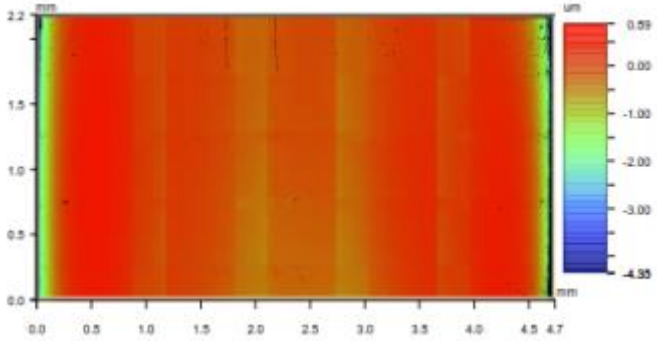
Structure relaxation overtime so characterization post irradiation needs to be planned accordingly!

Surface profile after annealing at 400°C for 8h

Prior to HT



After HT



Zn2.5	Ra (nm)	Rq (nm)	Rz (nm)	Rt (nm)
Prior to heat treatment				
non-irradiated area	38	46	431	505
irradiated spot	121	154	621	642
After heat treatment				
non-irradiated area	**66**	**76**	**319**	**336**
former irradiated spot	**162**	**191**	**897**	**926**

After HT

- Spots are not visible anymore
- No change in the surface roughness