DE LA RECHERCHE À L'INDUSTRIE

RESTRUCTURATION DES VERRES NUCLÉAIRES SOUS L'EFFET DES DÉSINTÉGRATIONS ALPHA

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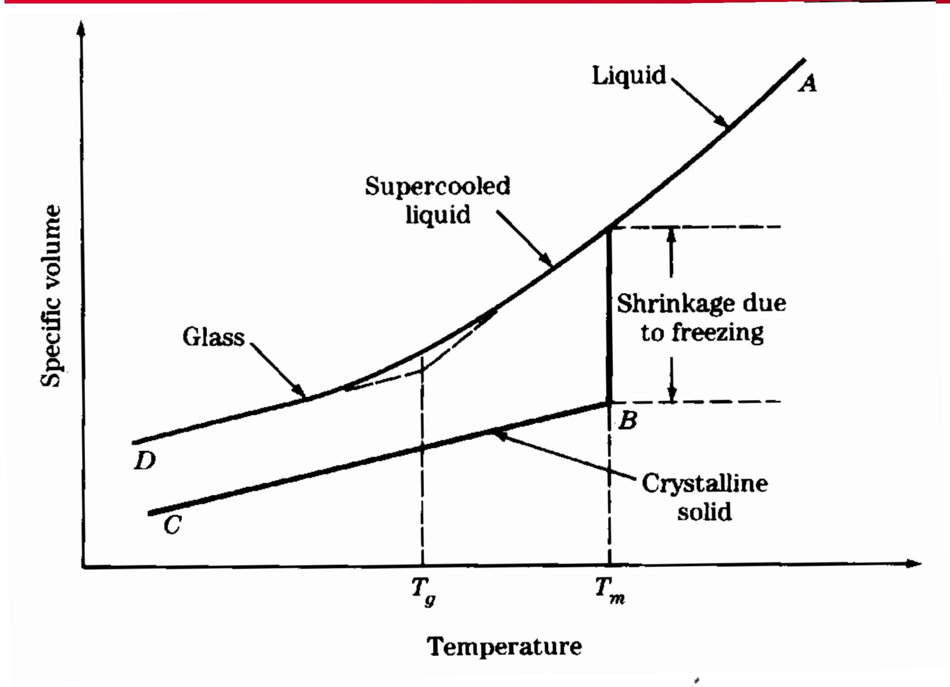
EURACT-nmr

LCV Joint Vitrification Lab

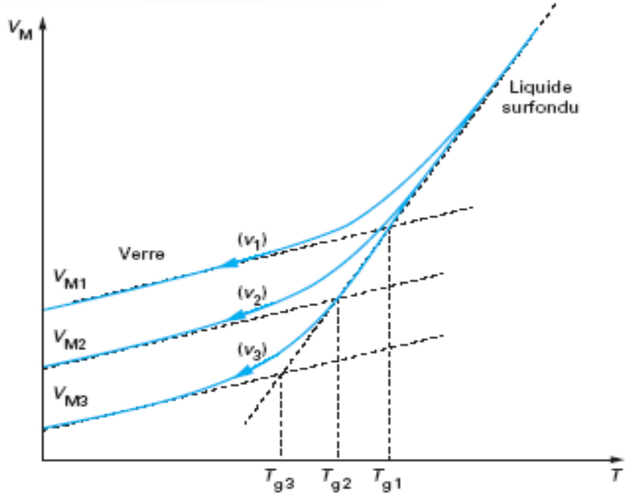
AREVA

Glassy state



A glass (or vitreous solid) is a solid formed by rapid melt quenching.

A glass is an amorphous solid that exhibits a glass transition phenomena at T_g.



V_{M1}, V_{M2}, V_{M3} indiquent les volumes molaires respectifs des verres obtenus pour les différentes vitesses de refroidissement :
$v_1 > v_2 > v_3$

(b) influence de la vitesse de refroidissement

Viscosity

$$\text{Relaxation time} \quad \tau = \frac{\eta}{G}$$

Shear Modulus

Glass properties depend on:

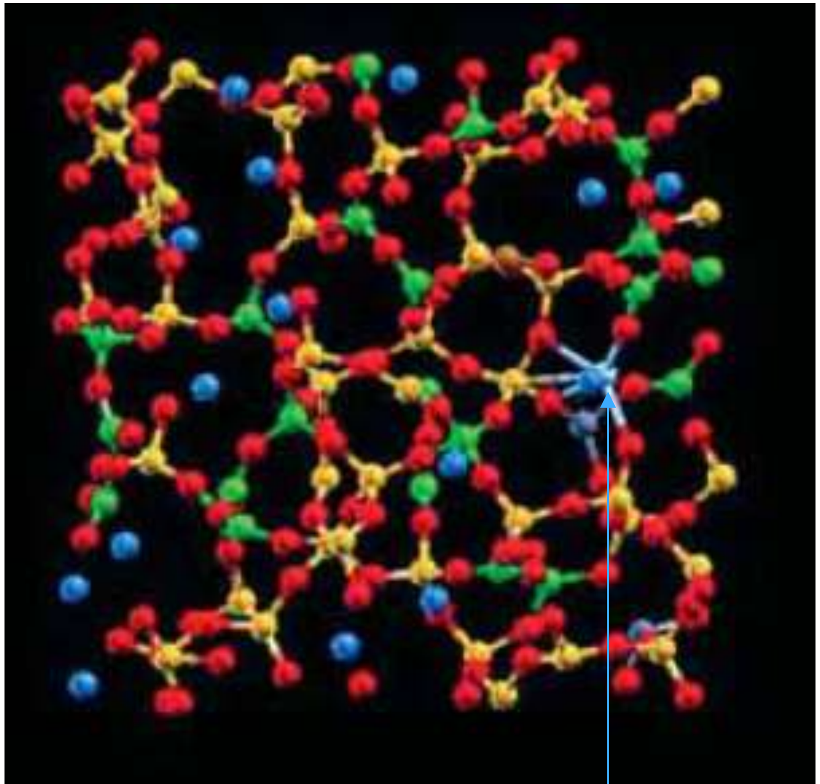
- **Chemical composition**
- **Thermal history during elaboration process**

Nuclear Glass: SON68

Oxide glass with around 30 oxides

Sodium alumino-borosilicate glass

L. Cormier, J.M. Delaye, D. Ghaleb, G. Calas, PRB 61 (2001) 14495



Si B Na
O

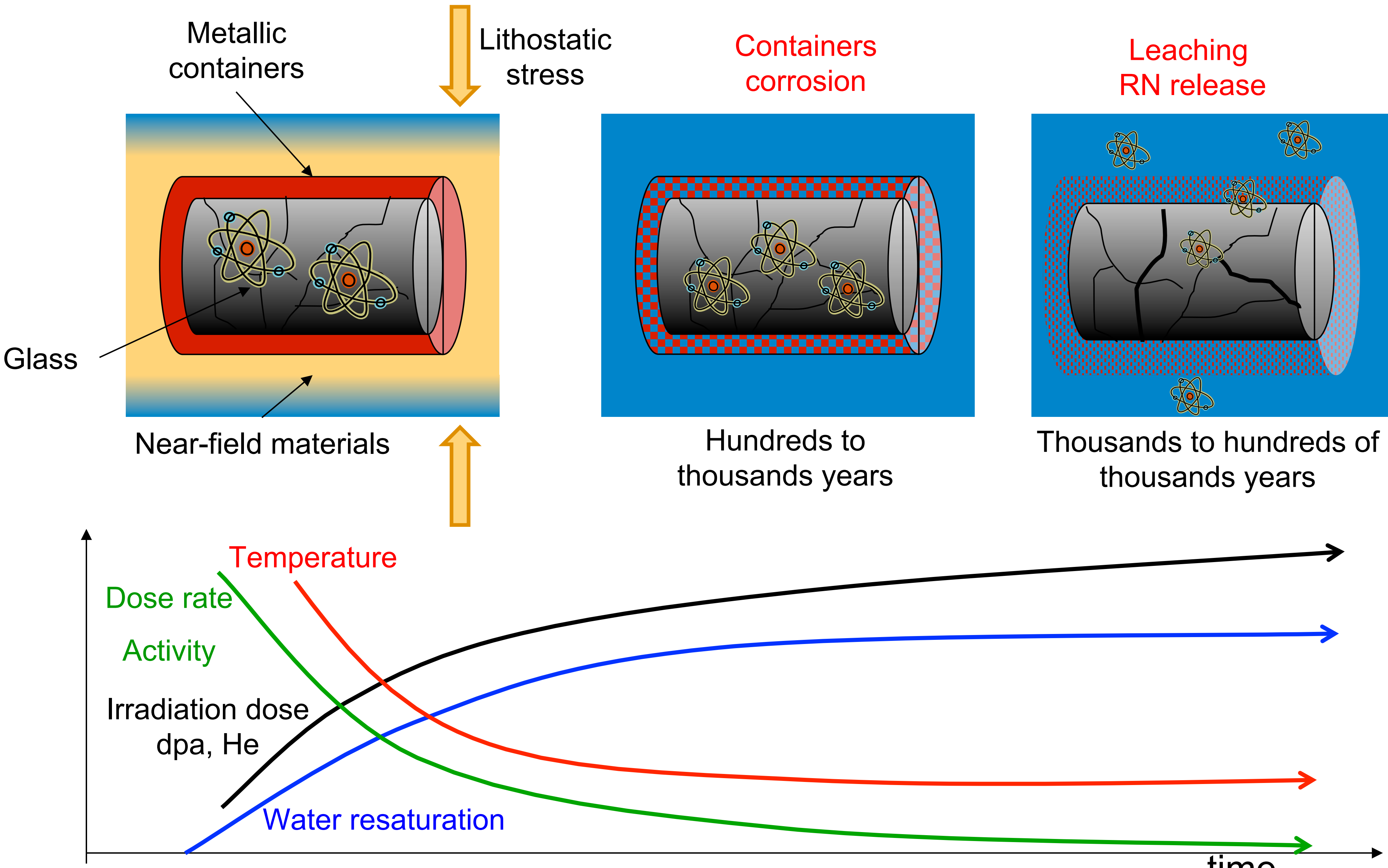
Fission product / Actinide in an octahedric site

Domaine de composition chimique des verres R7T7 produits dans les ateliers industriels par AREVA - La Hague

Oxydes	Intervalle spécifié pour l'industriel (% massique)		Composition moyenne des verres industriels (% massique)
	min	max	
SiO_2	42,4	51,7	45,6
B_2O_3	12,4	16,5	14,1
Al_2O_3	3,6	6,6	4,7
Na_2O	8,1	11,0	9,9
CaO	3,5	4,8	4,0
Fe_2O_3		< 4,5	1,1
NiO		< 0,5	0,1
Cr_2O_3		< 0,6	0,1
P_2O5		< 1,0	0,2
Li_2O	1,6	2,4	2.0
ZnO	2,2	2,8	2,5
Ox (PF+Zr+ actinides)+ Suspension de fines	7,5	18,5	17,0
Oxydes d'actinides			0,6
$SiO_2+B_2O_3+Al_2O_3$	> 60		64,4

Monographie DEN : Le conditionnement des déchets nucléaires

Glass Long Term Behavior



Outline

1- Context: radiation sources

2- Methodology to study alpha decays impact

3- Effects of α decays on the glass properties and structure

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1- Context: radiation sources

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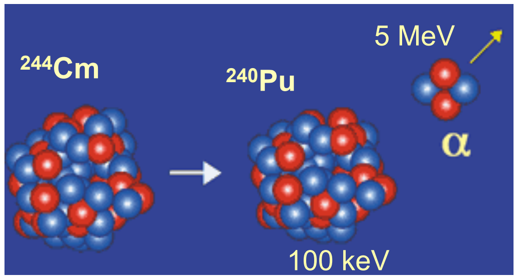
Glass LTB: What type of radiation?

Commercial glass

Irradiation source	path	Deposited energy (Gy)		Number of atomic displacements per event	Number of event per gram of glass after 10^4 years	Number of atomic displacements after 10^6 years
		After 10^4 years	After 10^6 years			
α decay **He (4 to 6 Mev)**	**~ 20 µm**	**~ $3x10^9$**	**~ 10^{10}**	**~ 200**	~ $3x10^{18}$	~ $6x10^{20}$
Recoil Nuclei (0.1Mev)	**~ 30 nm**	~ $6x10^7$	~ $3x10^8$	**~ 2000**		**~ $6x10^{21}$**
β decay	**1mm**	~ $3x10^9$	~ $4x10^9$	**~1**	$7x10^{19}$	$7x10^{19}$
γ transition	**qqs cm**	~ $2x10^9$	~ $2x10^9$	**<< 1**	$2x10^{19}$	<<$2x10^{19}$
(α, n) reactions	1m	~ $2x10^2$	~ $9x10^3$	200 à 2000	$3x10^{12}$	$6x10^{14}$ à $6x10^{15}$
Spontaneous Fissions	Ffs : 10µm neutron: 1m	~ $2x10^4$	~ $4x10^4$	10^5 200 à 2000	10^{11} à 10^{12}	10^{16} à 10^{17} $2x10^{13}$ à 10^{15}

Alpha decay

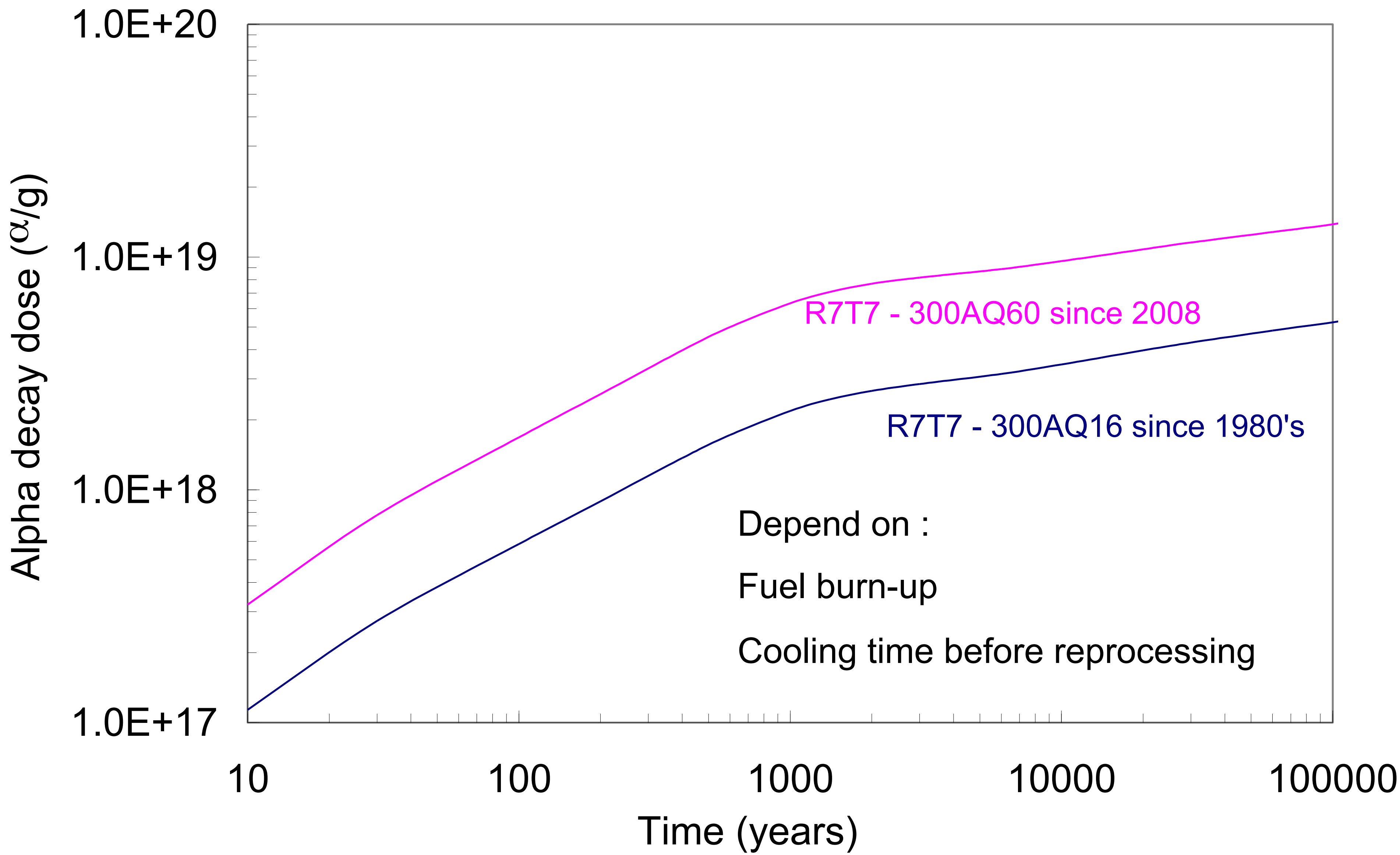


Minor actinides:

- Main source of atomic displacements in the glass structure
- Main source of energy deposition over long term

What is the effect of alpha self-irradiation on the glass long term behavior?

1- Level of alpha decay dose ? Commercial HLW glass



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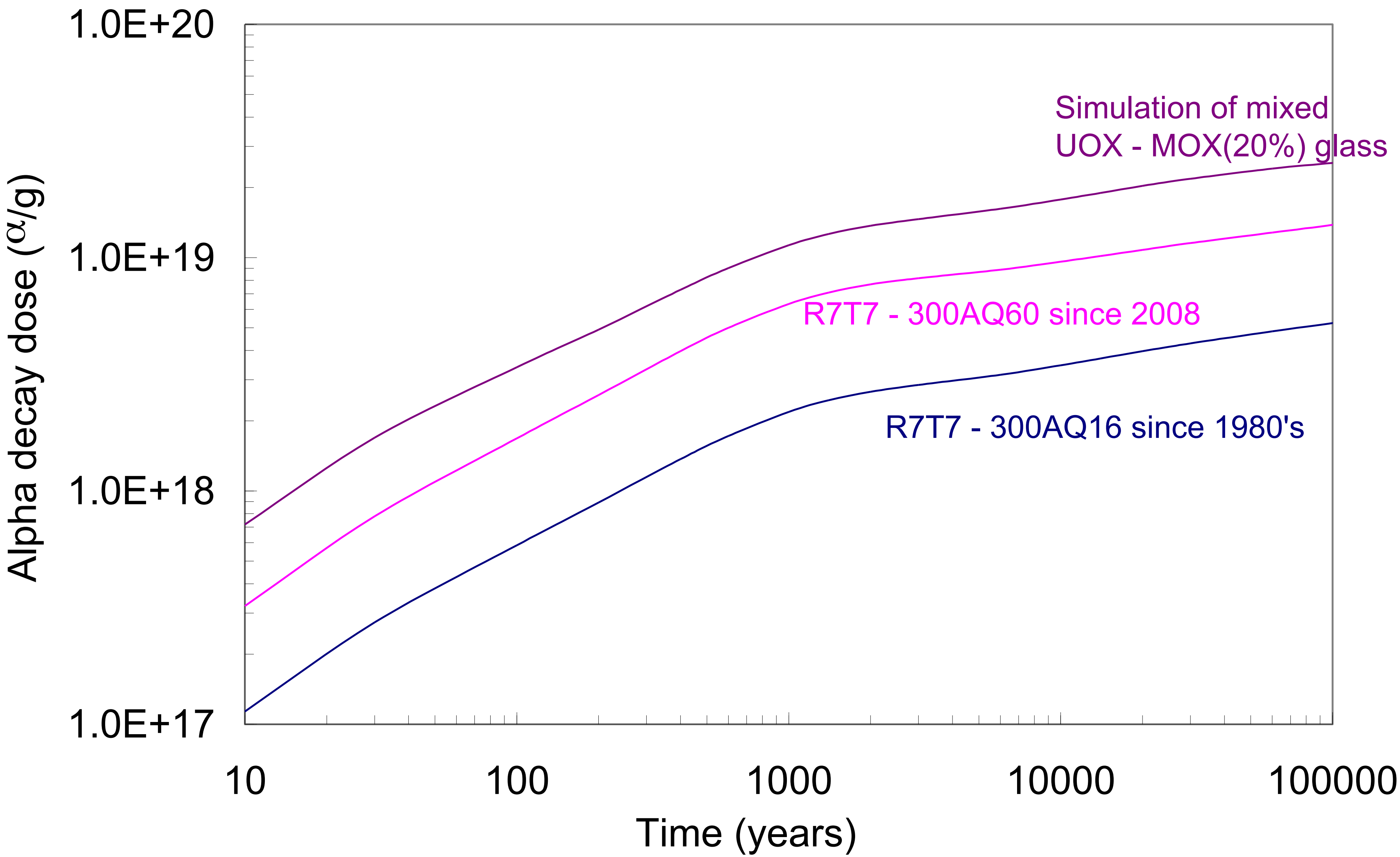
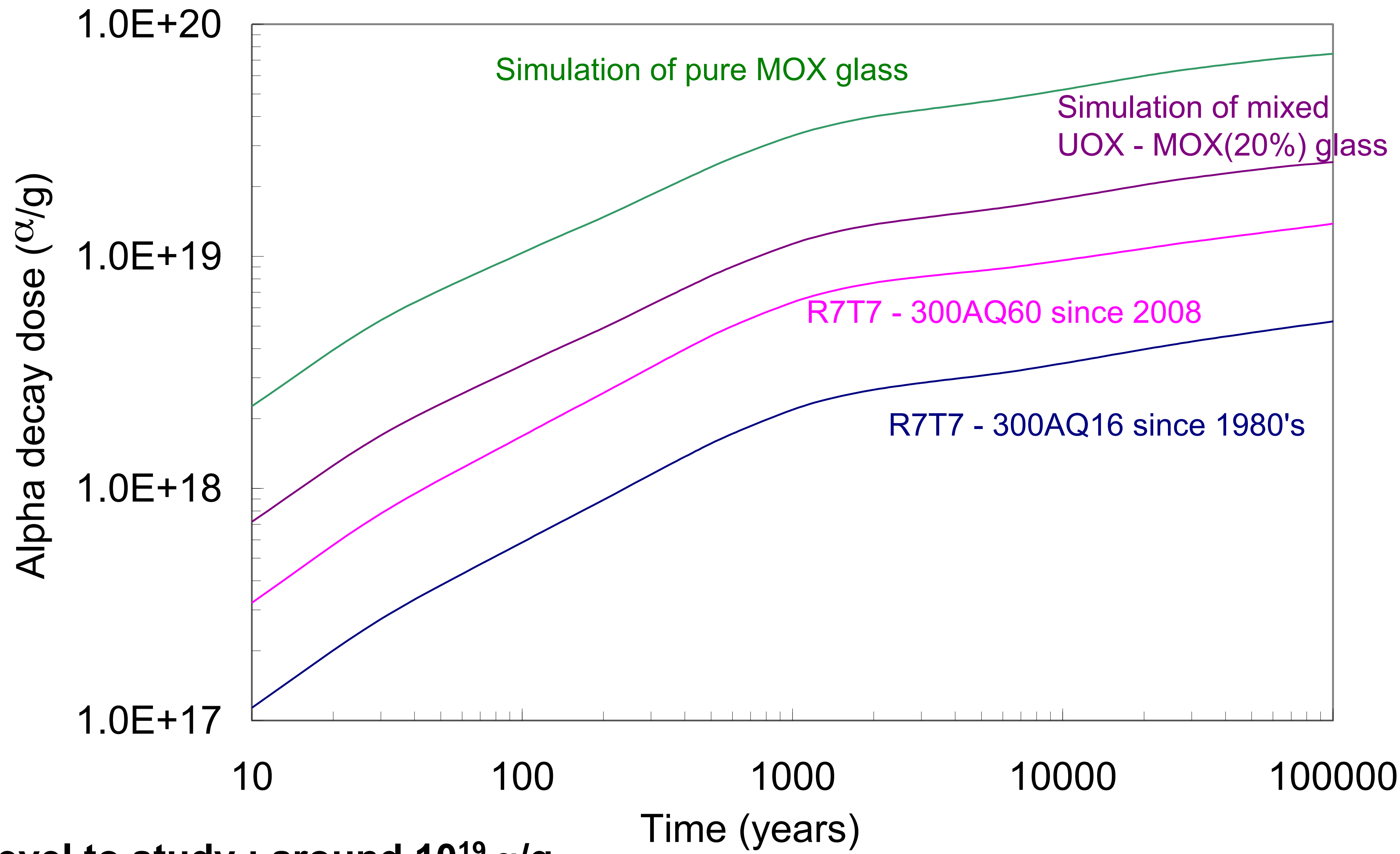
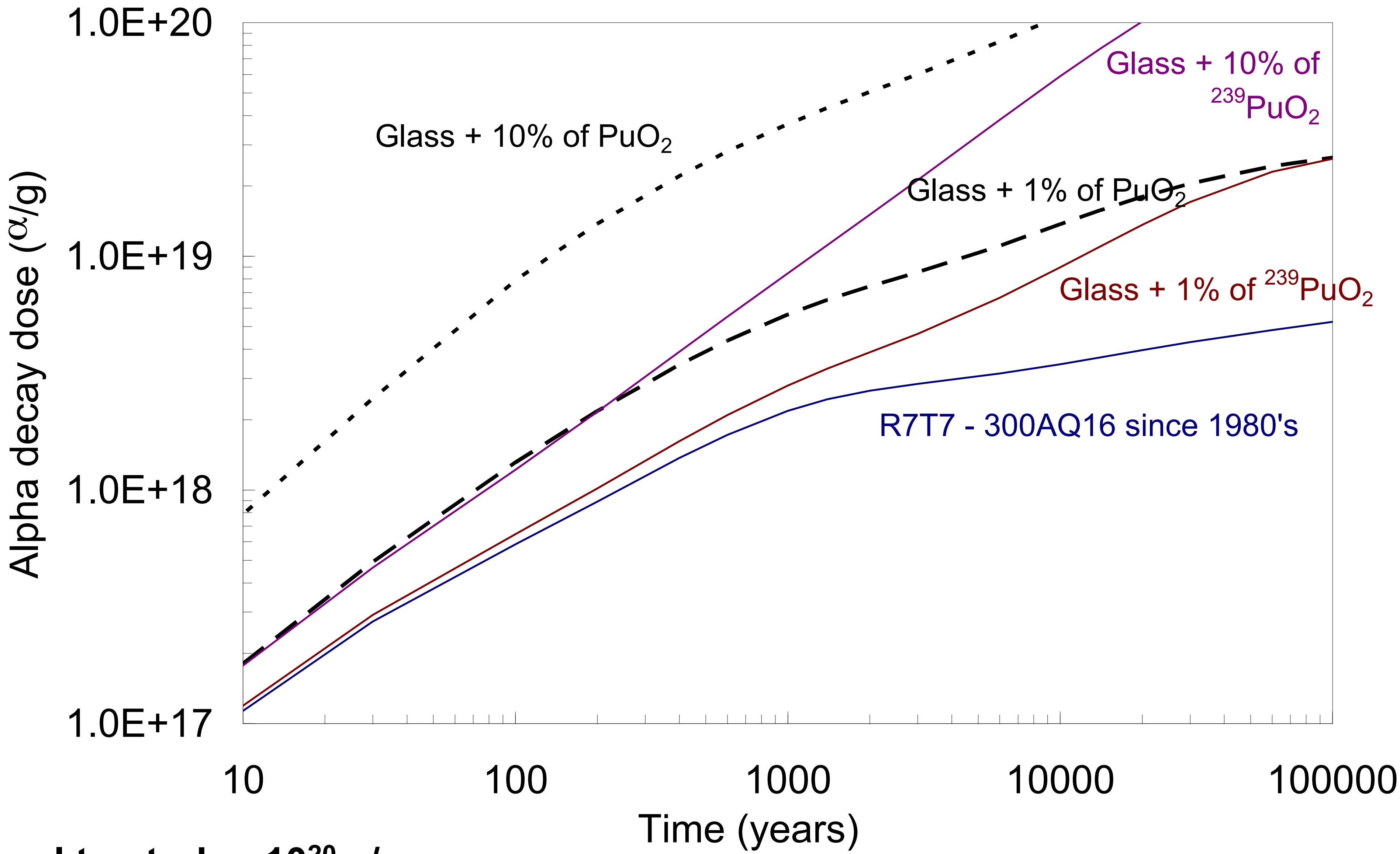


1- Level of alpha decay dose ? Commercial HLW



Level to study : around 10^{19} α/g

1- Level of alpha decay dose ? Pu immobilization



Level to study : 10^{20} α/g

1- Main studies on alpha decays effects

Main laboratory studies of alpha decay impact

USA (NLs)	70's-90's	<3 x 10^{18} α/g
UK (AERE)	70's-80's	<3 x 10^{18} α/g
France (CEA)	70's- 80's	<3 x 10^{18} α/g
EU (ITU)	70's-90's	<5 x 10^{18} α/g
JAPAN (JAERI)	90's	<10^{19} α/g

Macrosocpic behavior but very few data on the glass structure !

Need to improve the understanding of alpha decays effects

To predict long term behavior

To explore nuclear glass limits

To optimize the future glass composition

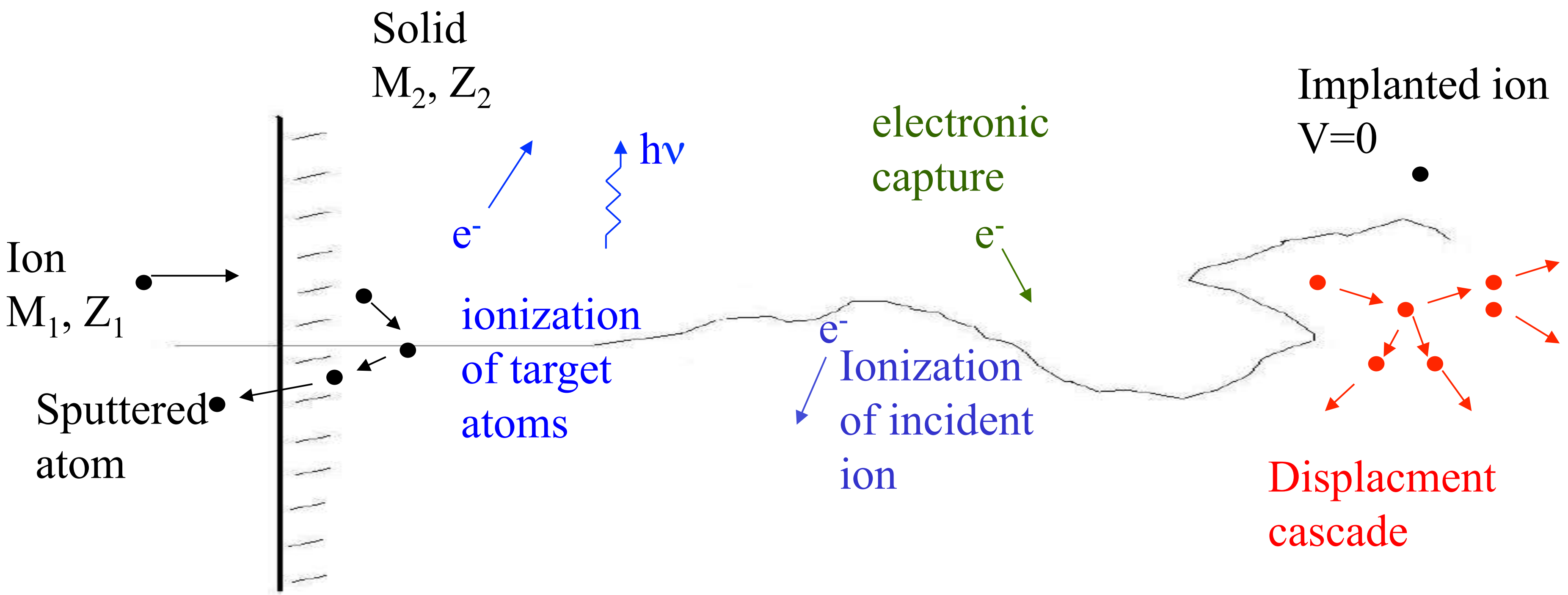
Focus on the results of the research program started in 2001 at CEA

Outline

1- Context: radiation sources

2- Methodology to study alpha decays impact

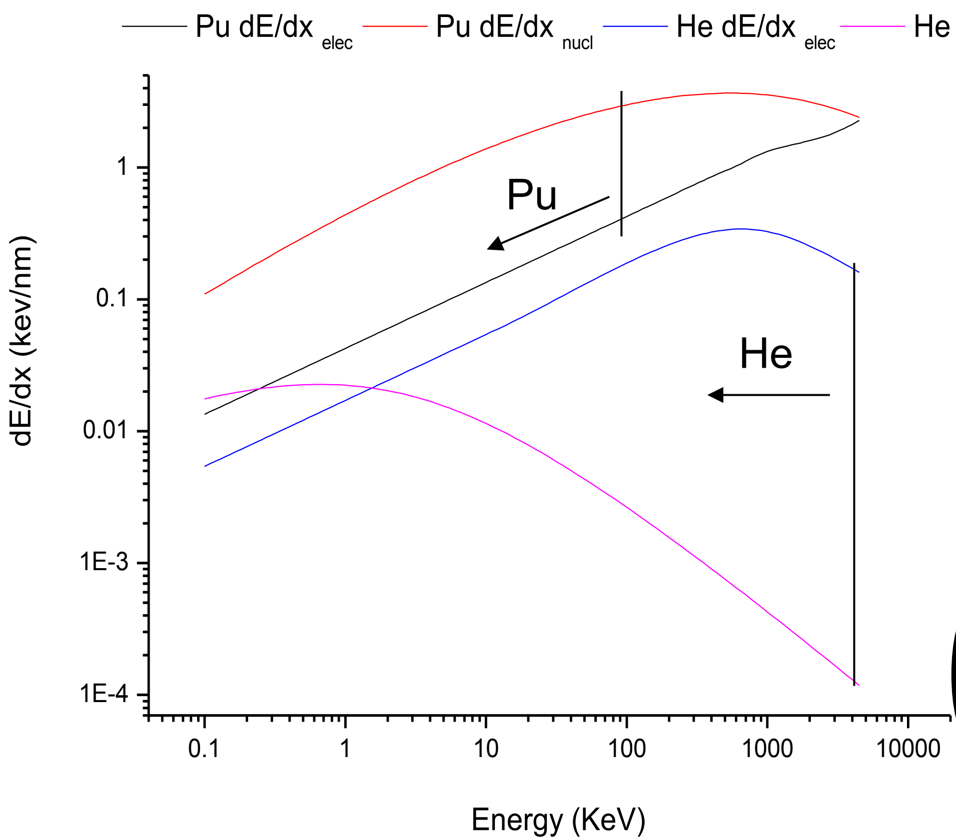
3- Effects of α decays on the glass properties and structure

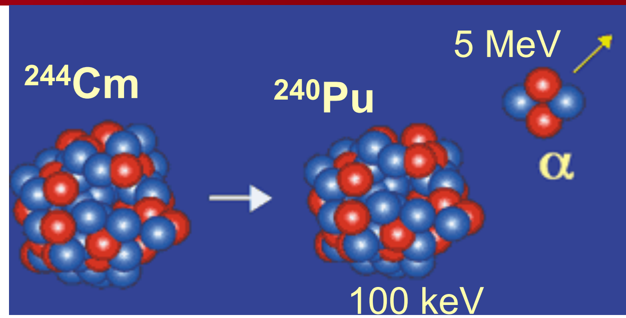


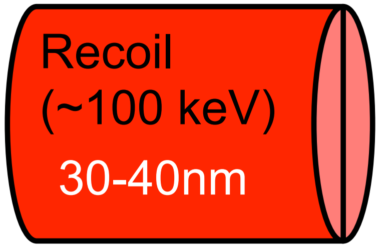
Electronic (inelastic) collision : electronic excitation / ionization of both partners of the collision event

Nuclear (elastic) collision : no modification of internal state of both partners of the collision event

2- Alpha decay

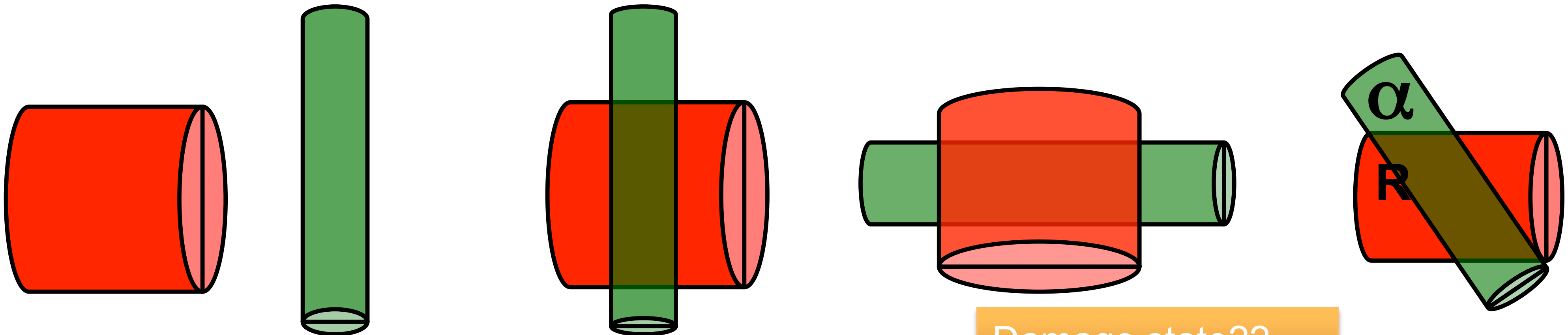






Mainly nuclear collisions
Ballistic damage
Displacement cascade

Mainly electronic collisions



Damage state??

2- Methodology to simulate alpha decays effects

- **Accelerate the time scale**
- **Dissociate the effects of self-irradiation (*<u>electronic / nuclear</u>*) and *<u>helium generation</u>***
- **Evaluate the effects on the confinement properties**
- **Evaluate the effects on the glass structure**

Propose some models to explain the glass behavior under alpha self-irradiation

1. Curium doped glasses

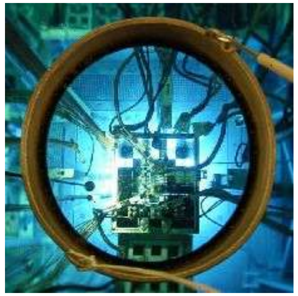
Atalante DHA, CEA

2. External irradiation with light and heavy ions

IPN Orsay Lyon, Ganil

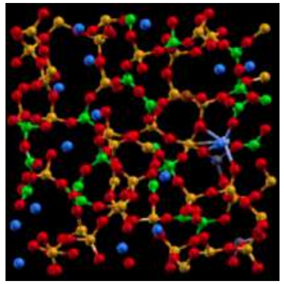
3. In pile irradiation : $^{10}B(n,\alpha)^{7}Li$

OSIRIS, CEA



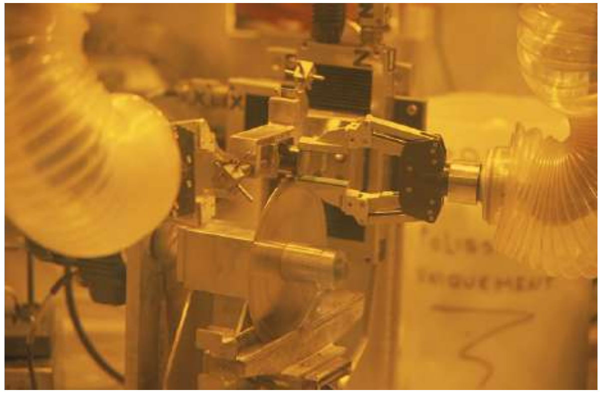
4. Molecular dynamic modeling of ballistic effects

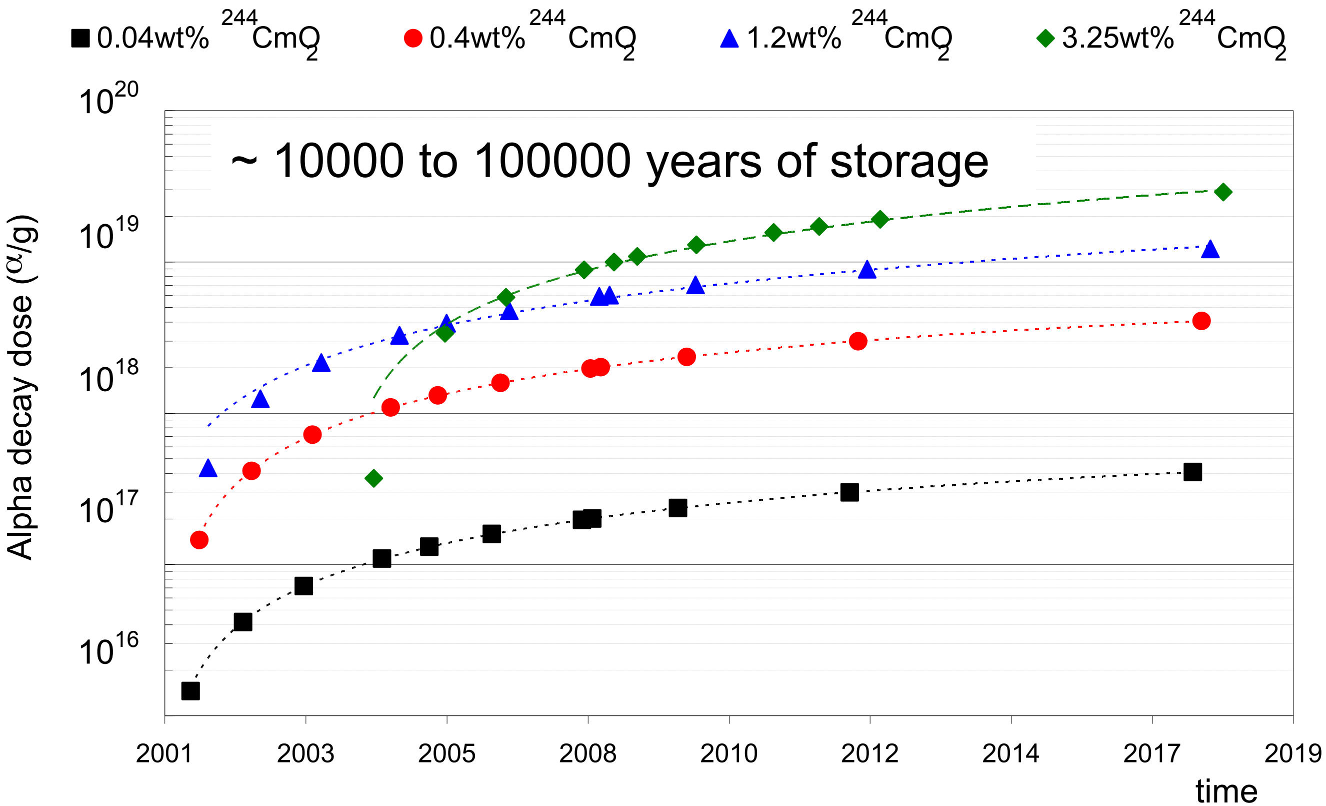
DM, CEA



2- Methodology: Cm doping

- SON68 glasses doped with 0.04, 0.4, 1.2, 3.25wt% of $^{244}CmO_2$
- Simplified glass (ISG) doped with 0.7wt% of $^{244}CmO_2$



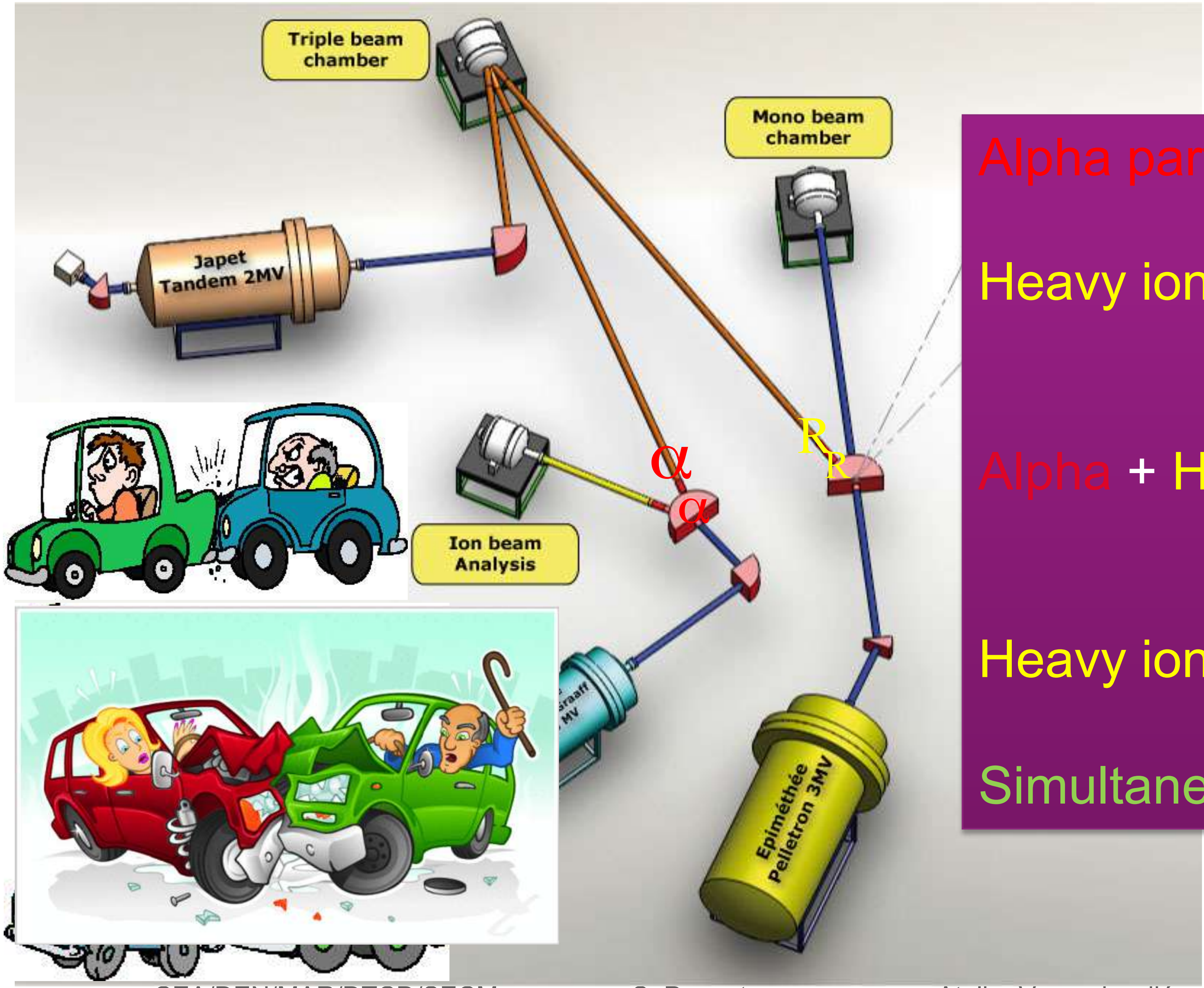


Mol%	SiO_2	Na_2O	B_2O_3	Al_2O_3	CaO	ZrO_2	Other oxides
ISG/CJ4	60.1	12.6	16.0	3.8	5.7	1.7	
R7T7	52.8	11.3	14.1	3.4	5.0	1.6	11.8

- Initial characterizations of the glasses (homogeneity, chemical composition)
- Periodical characterizations of the glass properties

2- Methodology: Ion beam irradiation experiment

Jannus Saclay,Orsay, Ganil



Alpha particles

Heavy ions

Mono beam

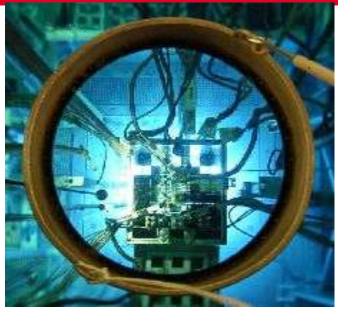
Alpha + Heavy ion

Heavy ion + Alpha

Simultaneous

Double beam

2- Methodology: In pile irradiation : $^{10}B(n,\alpha)^{7}Li$



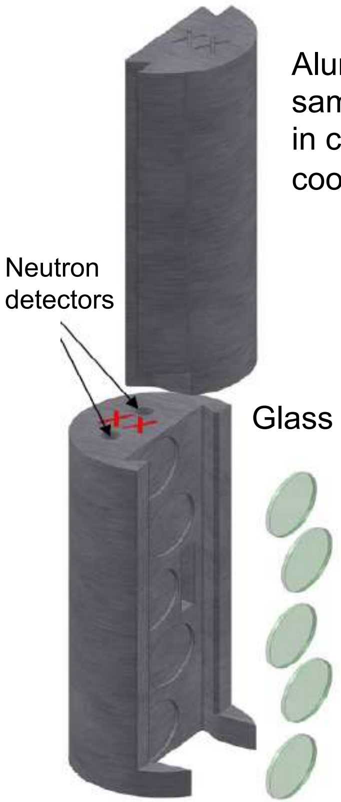
OSIRIS reactor, CEA SACLAY

	D1	D2	D3	D4
E (MeV)	He(1.47) + Li(0.84)			
Fluence (neutron cm^{-2})	5.9 ×10^{18}	1.2 ×10^{19}	3.5 ×10^{19}	5.2 ×10^{19}
Number of events (ion cm^{-3})	3.5 ×10^{19}	7.0 ×10^{19}	2.1 ×10^{20}	3.1 ×10^{20}
dE/dx$_{nucl}$ (keV nm^{-1})	dE/dx(He) <0.03 dE/dx(Li) <0.06			
dE/dx$_{elec}$ (keV nm^{-1})	dE/dx(He) <0.33 dE/dx(Li) <0.56			
E$_{nucl}$ (GGy)	0.06	0.13	0.39	0.57
E$_{elec}$ (GGy)	5.16	10.45	30.69	45.71
Dpa	0.27	0.54	1.6	2.38

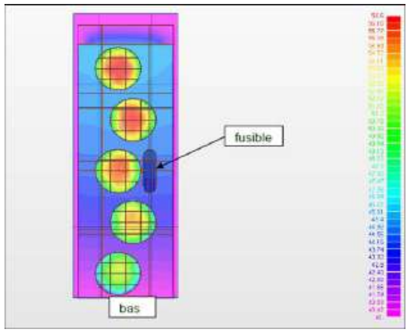
~ 1 billion years of disposal

Glass samples : polished disks thickness 0.5 mm

Mol%	SiO_2	Na_2O	B_2O_3	Al_2O_3	CaO	ZrO_2	Other oxides
CJ1	67.7	14.2	18.1				
SON68	52.8	11.3	14.1	3.4	5.0	1.6	11.8




Fuse



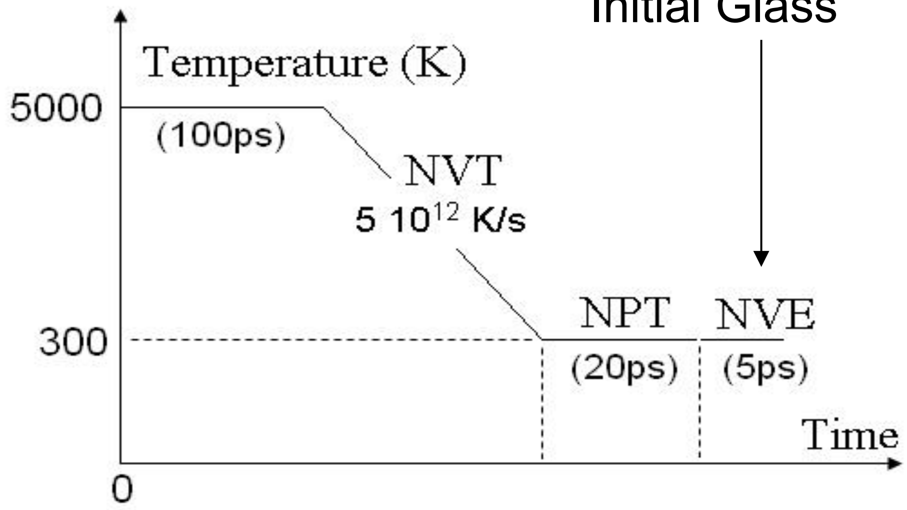
Thermal modeling and fuses observations after irradiation:

T<70°C

2- Methodology: Molecular dynamic modeling

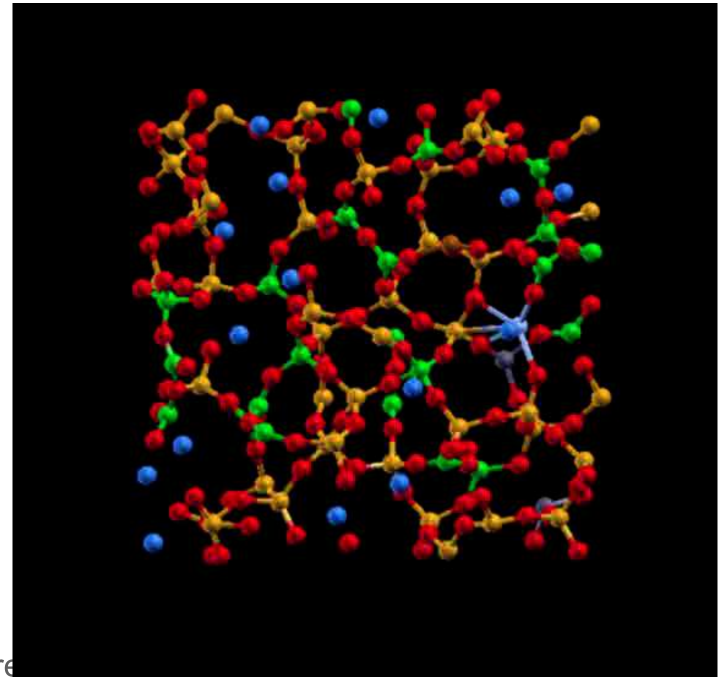
- Simplified borosilicate glasses (CJ1, CJ7)

$$\phi(r_{ij}) = \frac{q_i q_j}{r_{ij}} + B_{ij} \exp\left(-\frac{r_{ij}}{\rho_{ij}}\right) - \frac{C_{ij}}{r_{ij}^6}$$



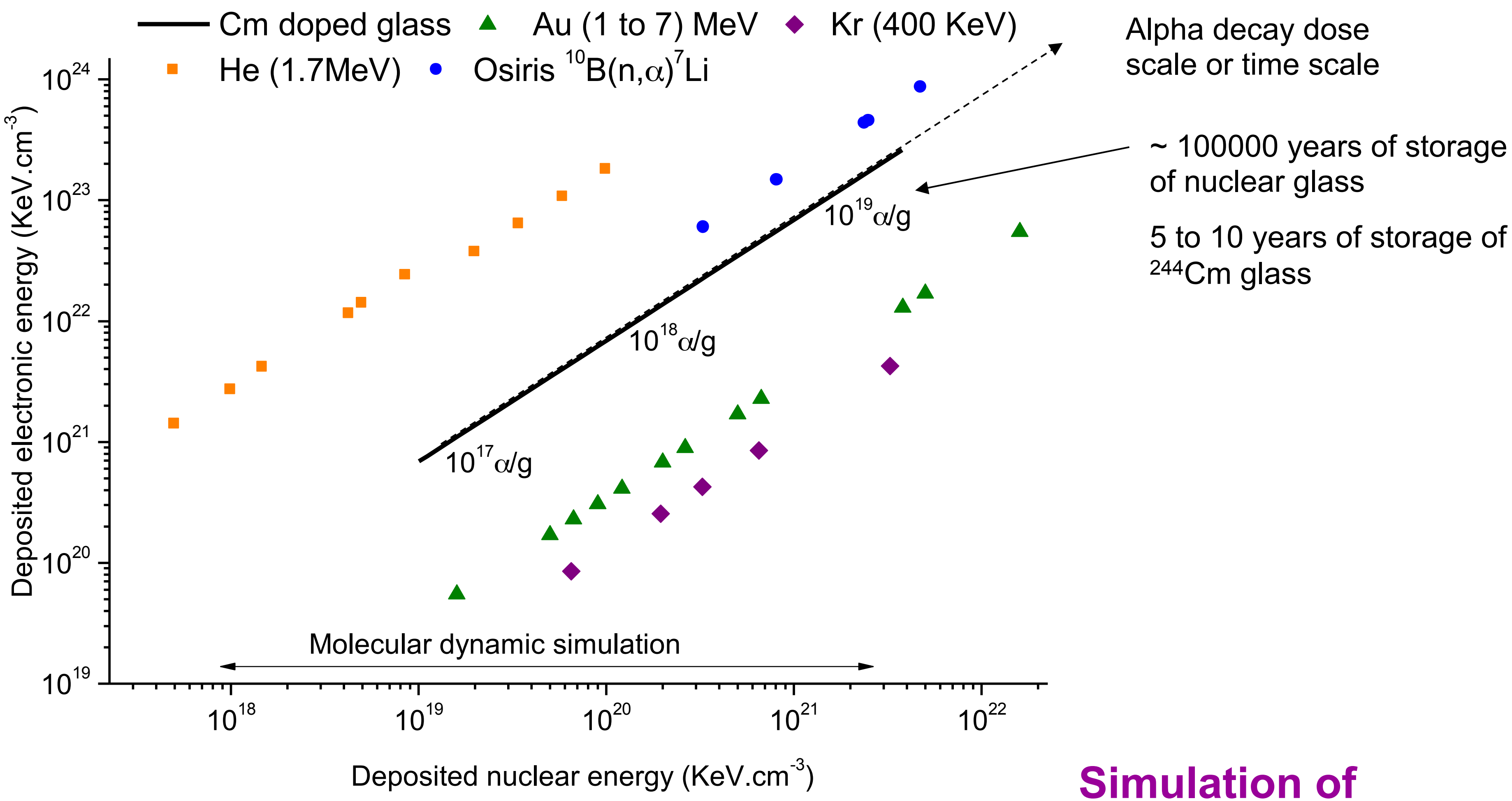
- Accumulation of displacement cascades caused by uranium atoms of energies from 700ev to 70keV

- Characterization of the structural modifications induced by displacement cascades



Mol%	SiO_2	Na_2O	B_2O_3	Al_2O_3	CaO	ZrO_2	Other oxides
CJ1	**67.7**	**14.2**	**18.1**				
CJ7	**63.8**	**13.4**	**17.0**	**4.1**		**1.8**	
SON68	*52.8*	*11.3*	*14.1*	*3.4*	*5.0*	*1.6*	*11.8*

2- Methodology: Materials and irradiation conditions



Light ions irradiations (He) : mainly electronic interactions

Heavy ions irradiations (Kr, Au) : mainly nuclear interactions

Doped glasses and OSIRIS irradiation : electronic and nuclear interactions

Molecular Dynamics : only nuclear interactions

Simulation of 100000 years of disposal by various methods !

Outline

1- Context: radiation sources

2- Methodology to study alpha decays impact

3- Effects of α decays on the glass properties and structure

3- Effect on the macroscopic properties? Density

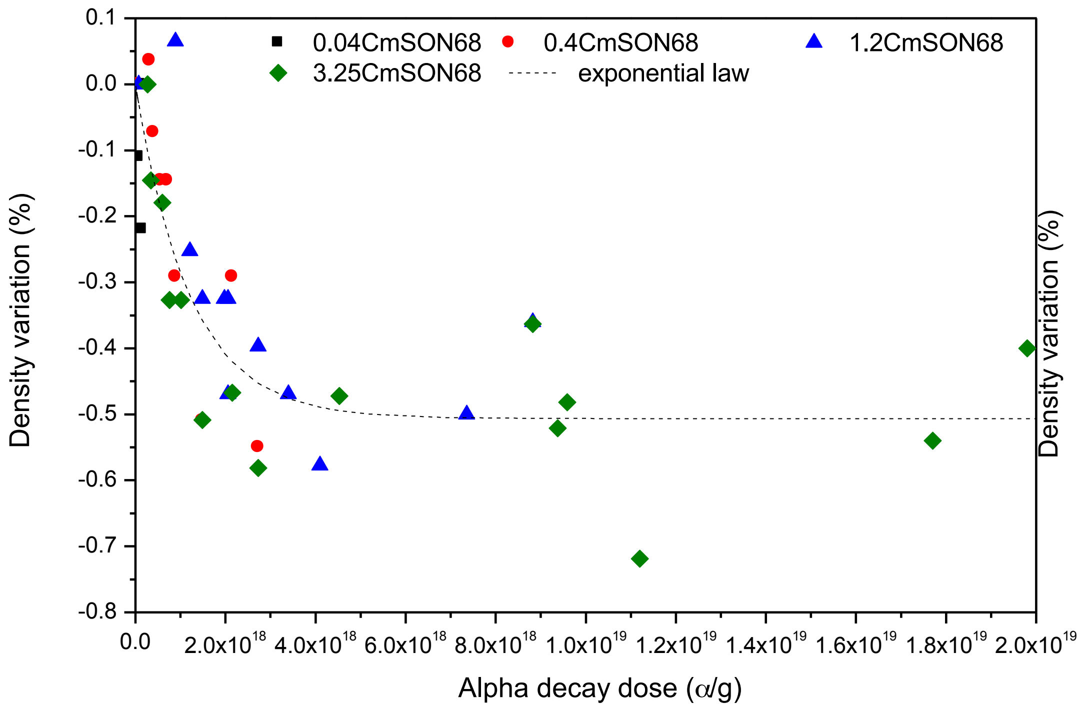
Slight decrease of the glass density (0.5%)

No effect of the dose rate

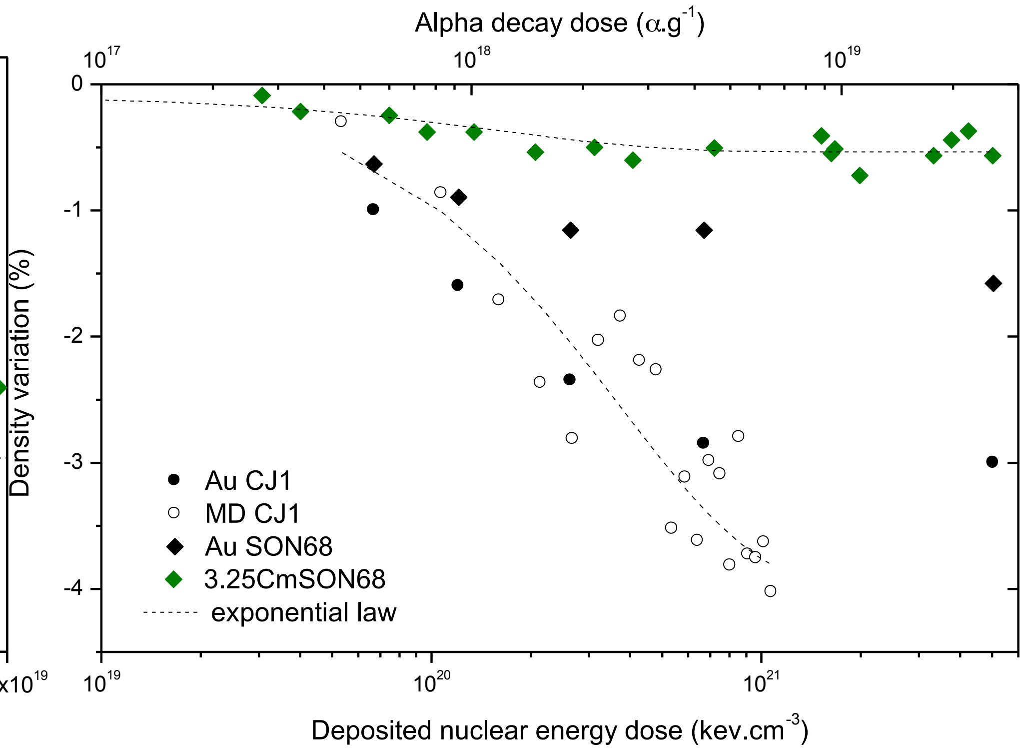
Stabilization of the evolution at around $4x10^{18}$ α/g

Evolution according to an exponential law (direct impact model)

- ✓Variations correclty simulated by external irradiations and MD simulation
- ✓Swelling level is lower under α decays irradiation (0,5% compared to 1,2% Au irradiation)



S. Peuget et al. J. Nucl. Mat. 354 (2006) 1



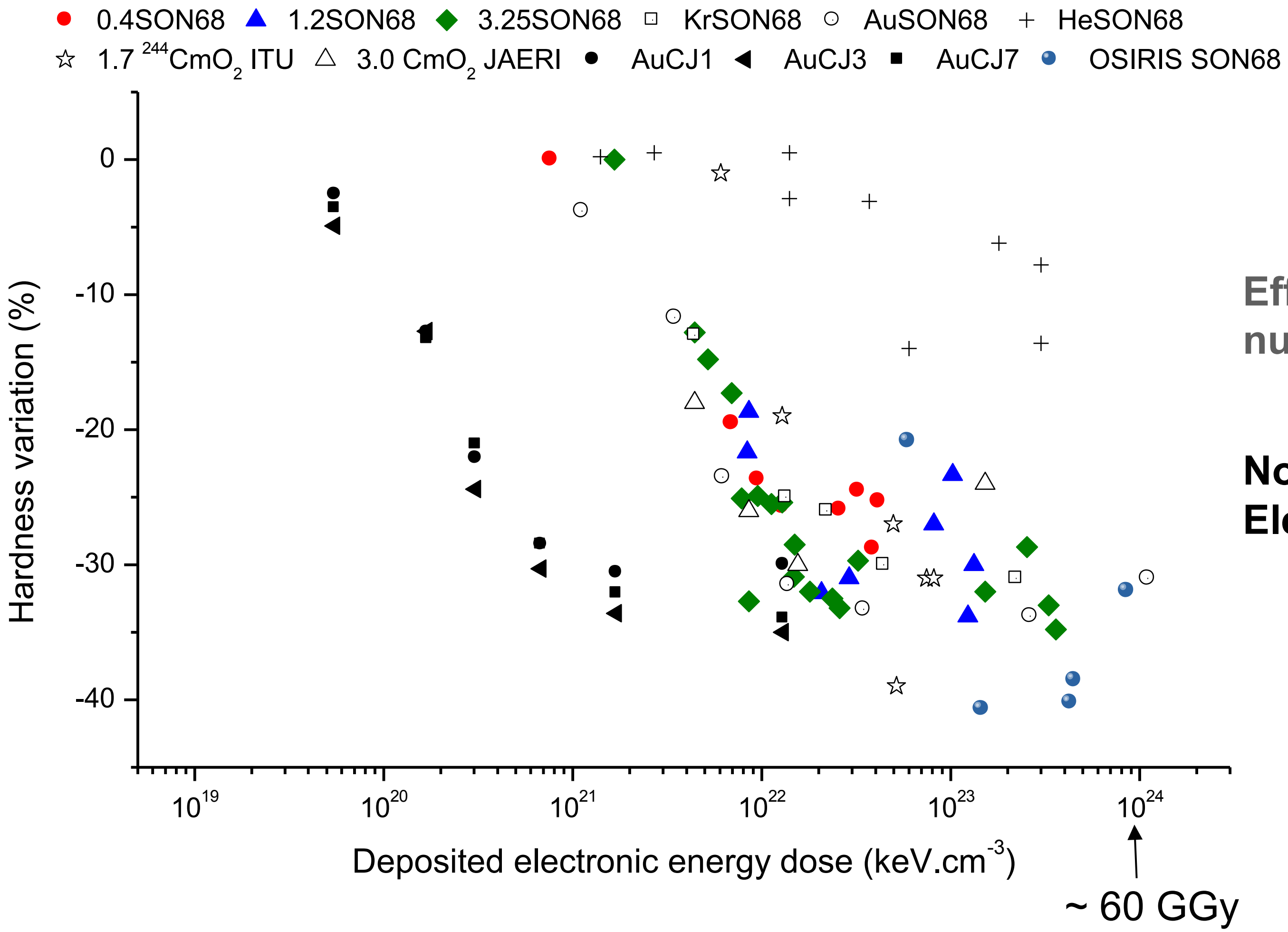
S. Peuget et al. J. Nucl. Mat. 354 (2014) 1

3- Effect on the macroscopic properties?

Mechanical properties: example of hardness

Decrease of hardness on curium doped glasses and ions irradiated glasses

He induced lower changes



Effect of electronic or nuclear interactions?

No agreement versus Electronic dose

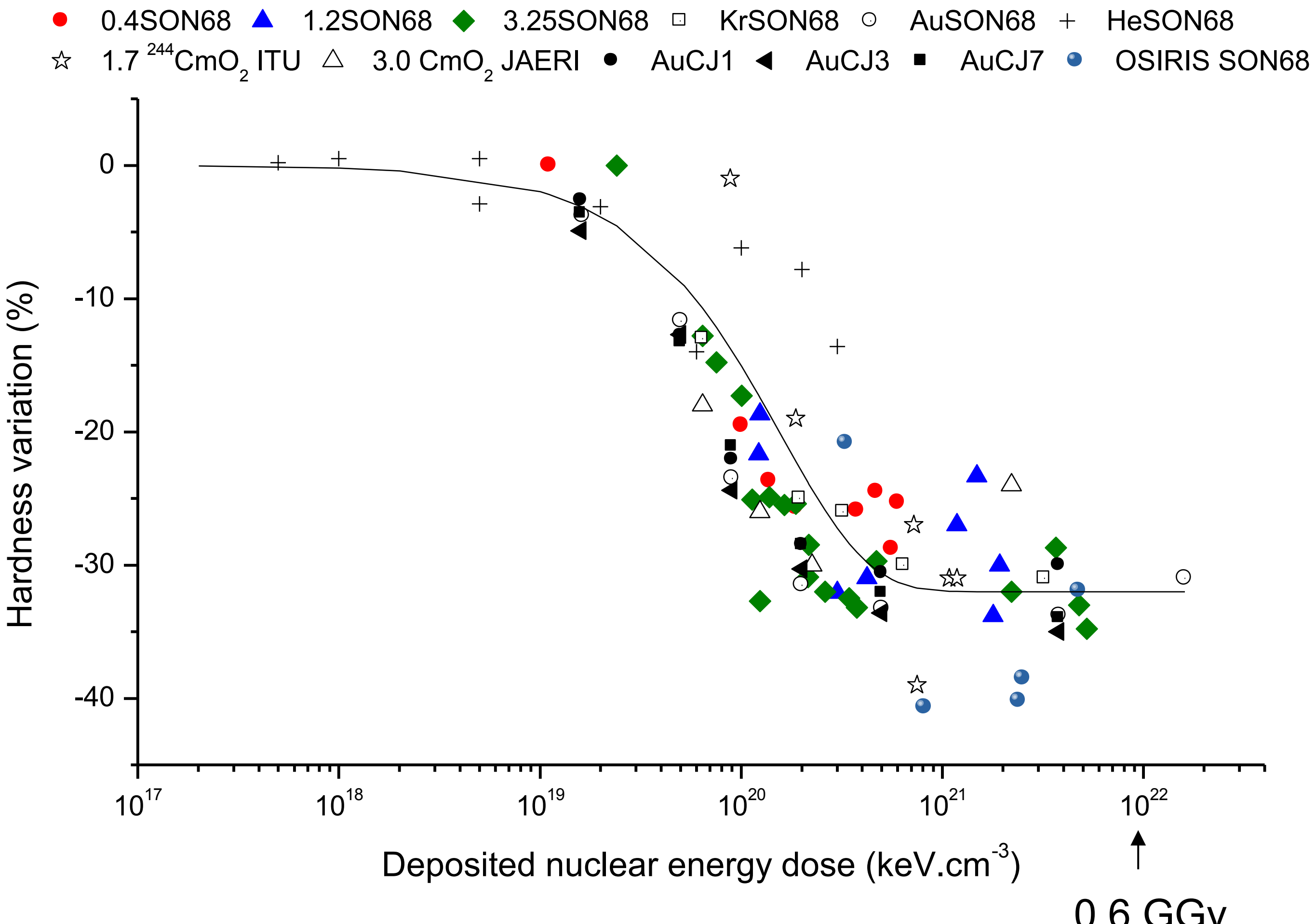
S. Peuget et al, NIMB 246 (2006) 379 S. Peuget et al. JNM 354 (2014) 1 Mir et al. . JNM, accepted

3- Effect on the macroscopic properties?

Mechanical properties: example of hardness

Decrease of hardness on curium doped glasses and heavy ions irradiated glasses

He induced lower changes



Effect of electronic or nuclear interactions?

Quite good agreement between doped glasses and heavy ions irradiated glasses

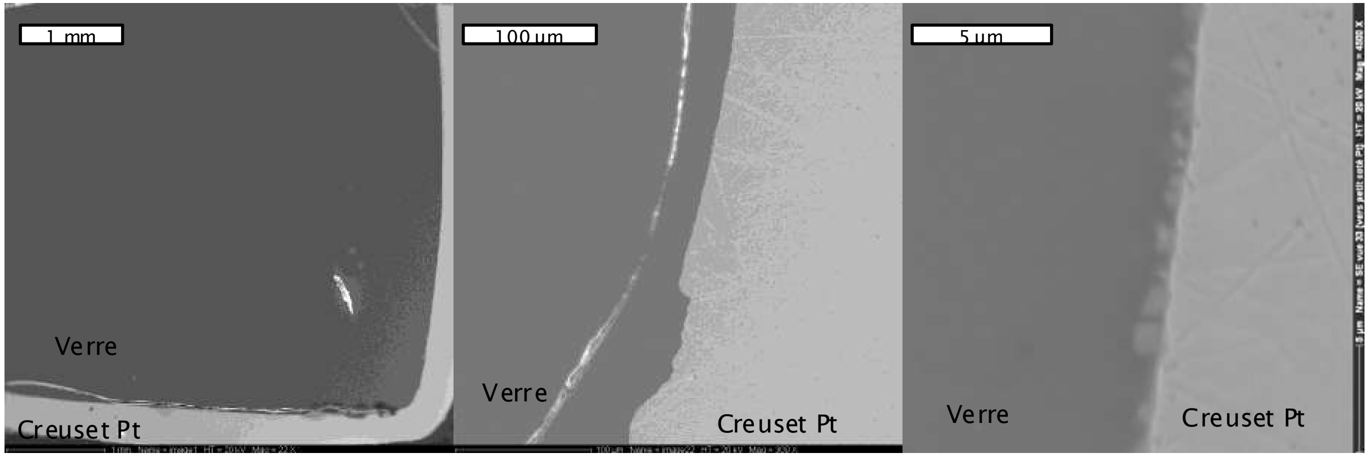
Effect induced by nuclear interactions

He case is different

S. Peuget et al, NIMB 246 (2006) 379 S. Peuget et al. JNM 354 (2014) 1 Mir et al. . JNM, accepted

3- Effect on the glass microstructure

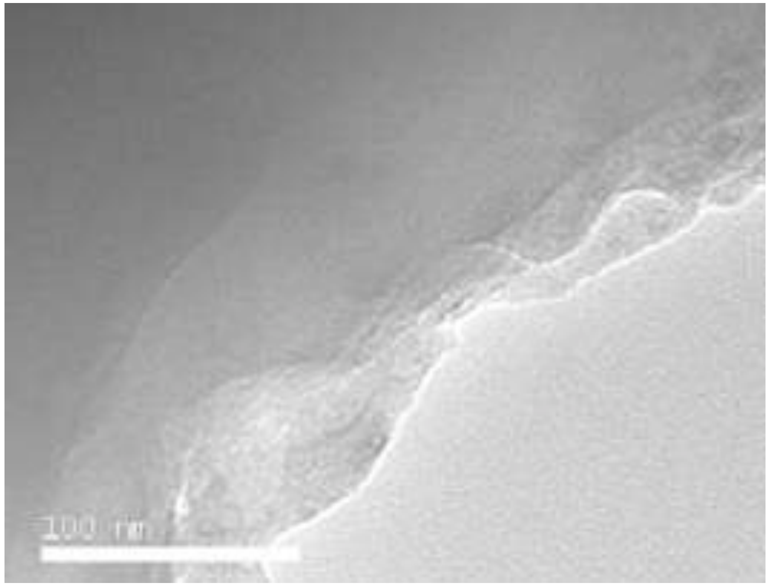
^{244}Cm SON 68 glass : SEM (CEA Marcoule), alpha decay dose 2x10^{19}α/g



(Around 100000 years of storage)

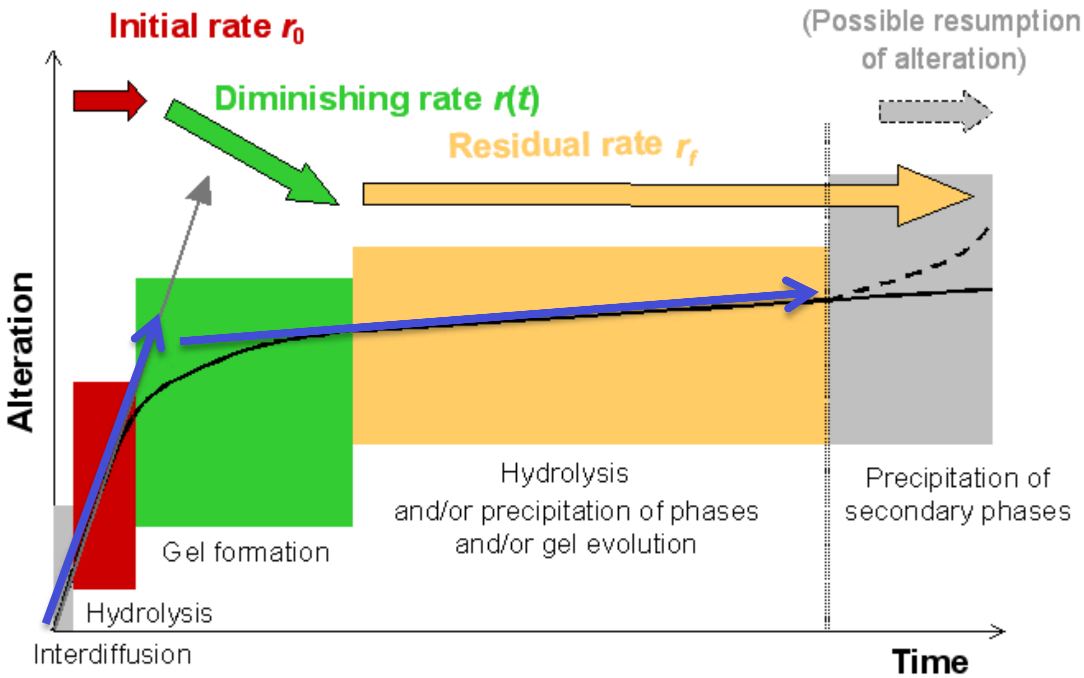
S. Peuget et al. JNM 44 (2014)

^{244}Cm SON 68 glass : TEM (ITU Karlsruhe), alpha decay dose 8x10^{18}α/g



Homogeneous microstructure, without bubbles, phase separation or crystallization

3- Effects of α radiation on the leaching behavior?



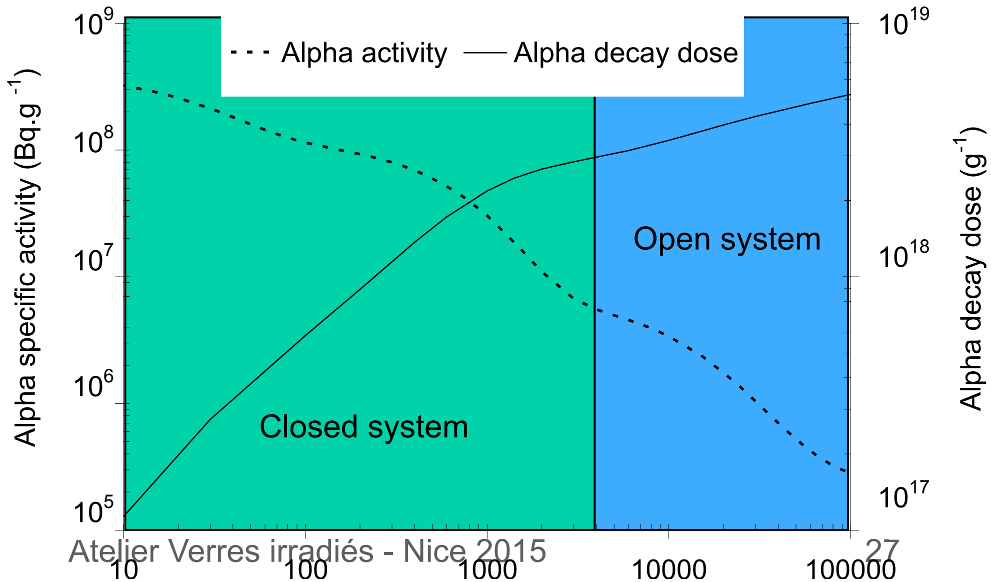
Two main steps to study:

1. **Impact on r_0**
2. **Impact on residual rate**

Two parameters to study:

1. **Dose rate**
2. **Dose**

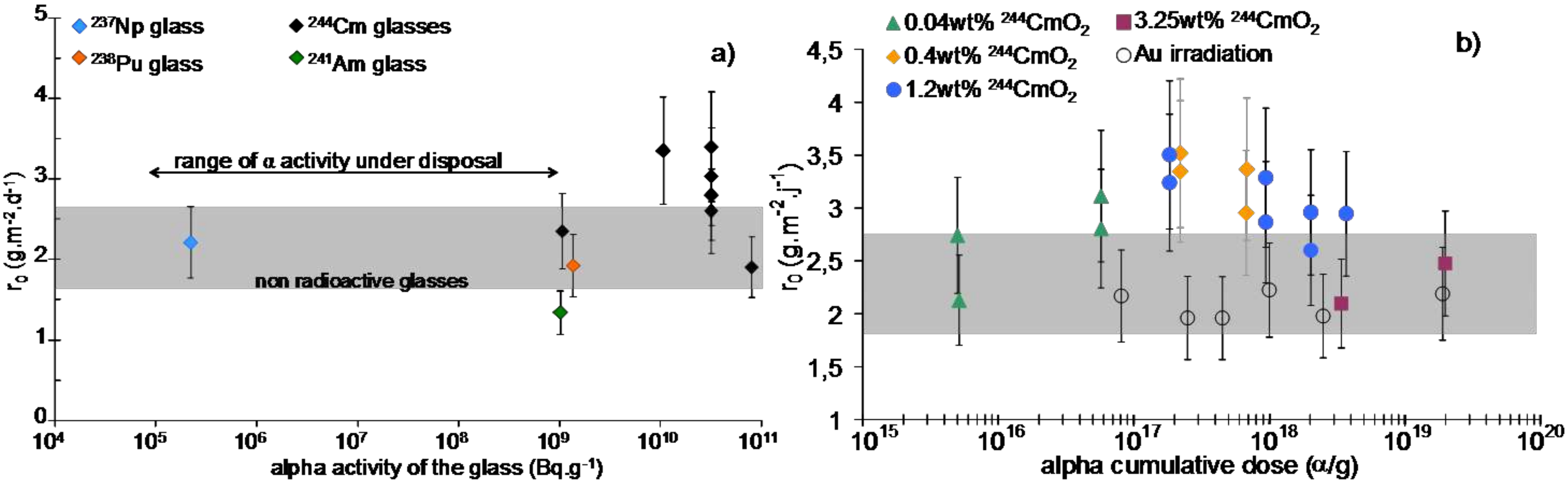
Experiments on alpha doped glasses and externally irradiated glasses



3- Effects of α radiation on the leaching behavior : r_o

Initial alteration rate, R_0: hydrolysis step, chemical reactivity with water

Soxhlet test with chemical analysis of the leachates



No significant effect of alpha dose rate on R_0

No significant effect of alpha decay dose or Au irradiation on R_0

S. Peuget et al. J. Nucl. Mat. 362 (2007) 374

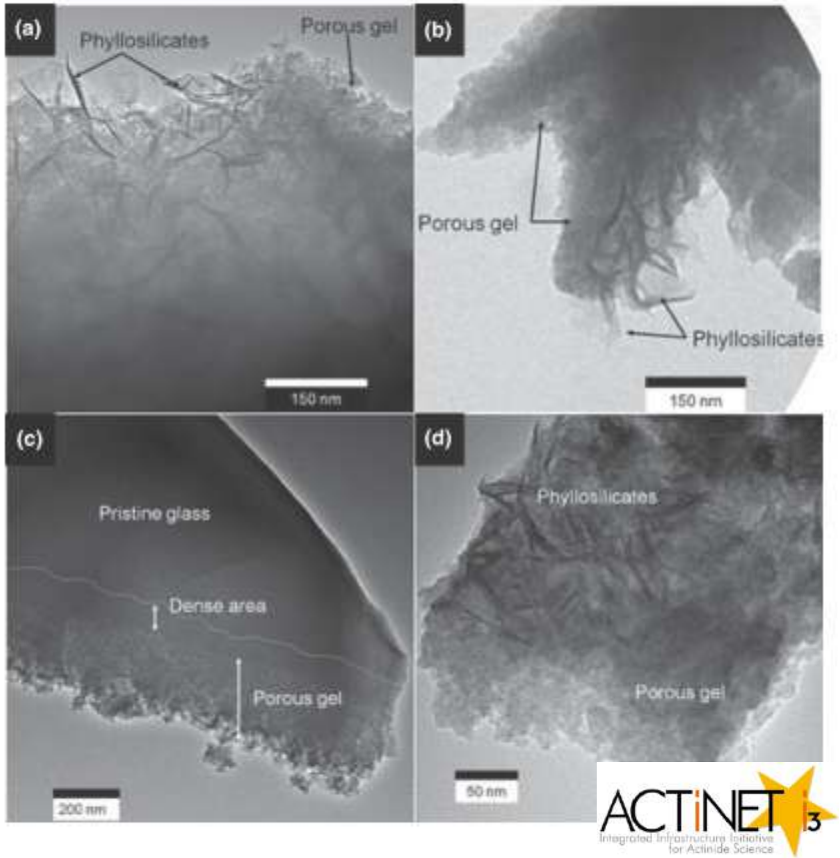
3- Effects of α radiation on the leaching behavior : r_r

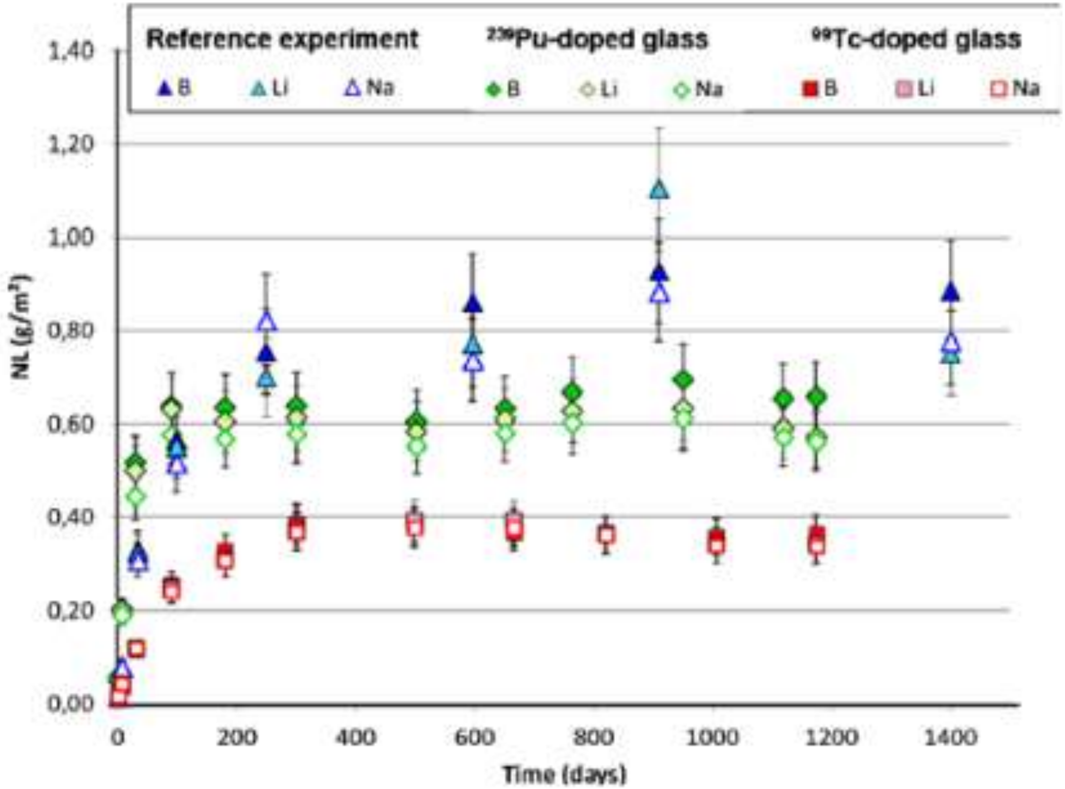
Residual rate, R_r ^{239}Pu glass dose rate ~ 1000 years of disposal

Static leaching test S/V=20cm^{-1}

TEM CEA

TEM ITU





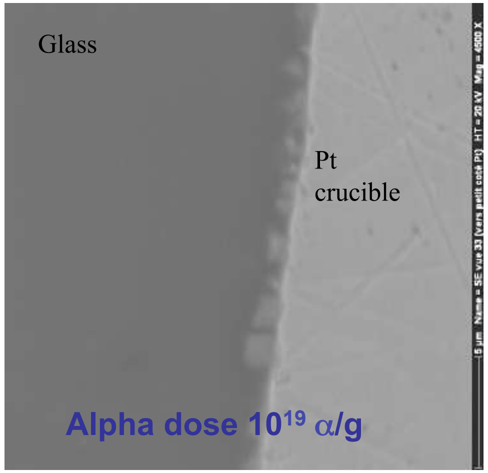
Similar alteration phenomenology with same R_r as for non-radioactive glass

Similar alteration products (PRI: phylosilicates, porous gel, dense area, pristine glass)

S. Rolland et al. Int. J. Appl. Glass Science 4 (2013) 295

No significant effect of alpha dose rate R_r

3- Summary on the macroscopic behavior: SON68 glass

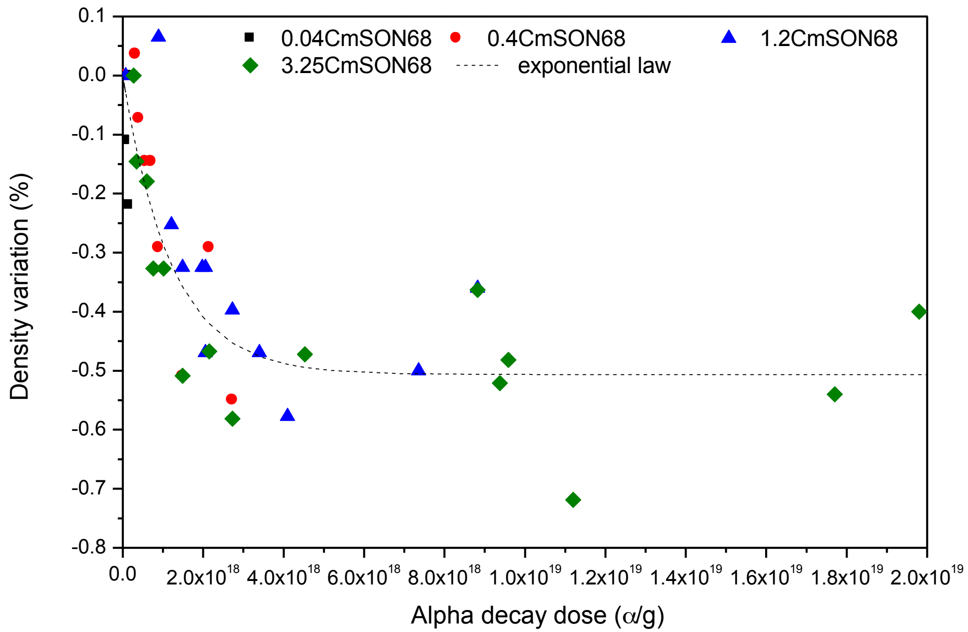


- **Slight decrease of the glass density (0.5%)**
- **Slight improvement of the mechanical properties (decrease of hardness and young modulus, increase of fracture toughness)**
- **Glass is still homogeneous (SEM and TEM scale)**
- **No effect on initial alteration rate**

Modifications observed in the first $4x10^{18}\alpha$/g

Effects induced by nuclear interactions (recoil nuclei)

No effect of the dose rate

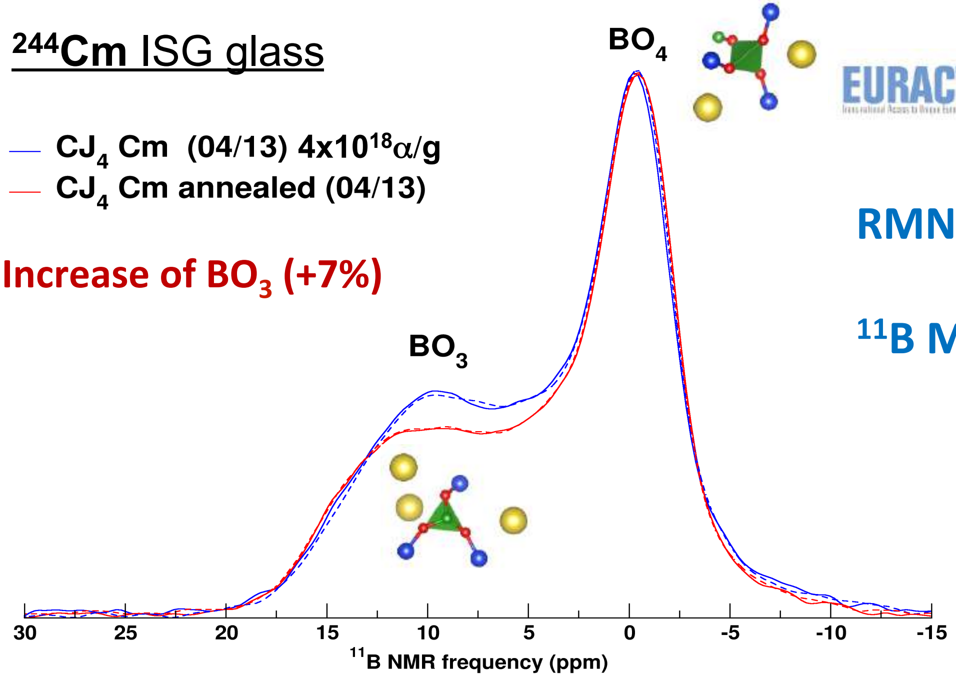


What are the structural origins of these modifications ?

Study of complex and simplified glasses ….

3- Effect on glass structure : SRO around B

^{244}Cm ISG glass



Increase of BO_3 (+7%)

T. Charpentier et al. in preparation

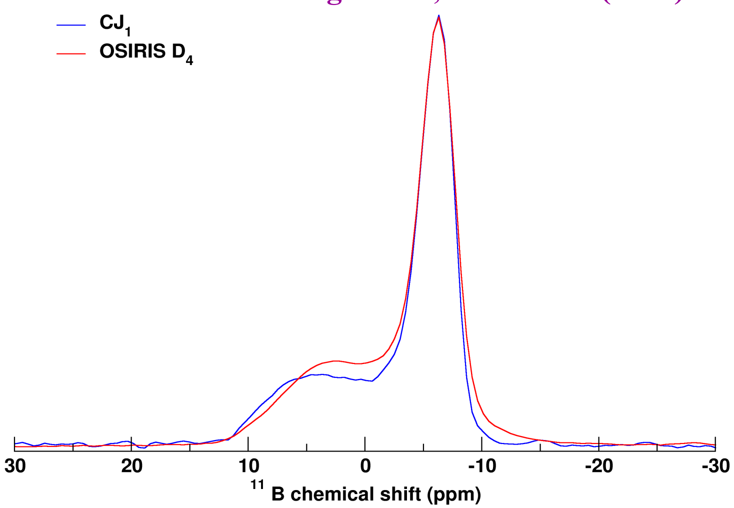
EURACT-nmr

RMN at ITU

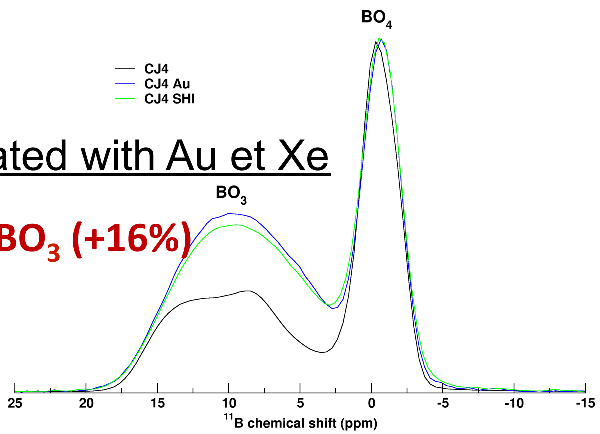
^{11}B MQMAS

CJ1 irradiated in OSIRIS reactor

S. Peuget et al, NIMB 327 (2014) 22-28



ISG irradiated with Au et Xe



Increase of BO_3 (+16%)

C. Mendoza et al. NIMB 325 (2014) 54-65

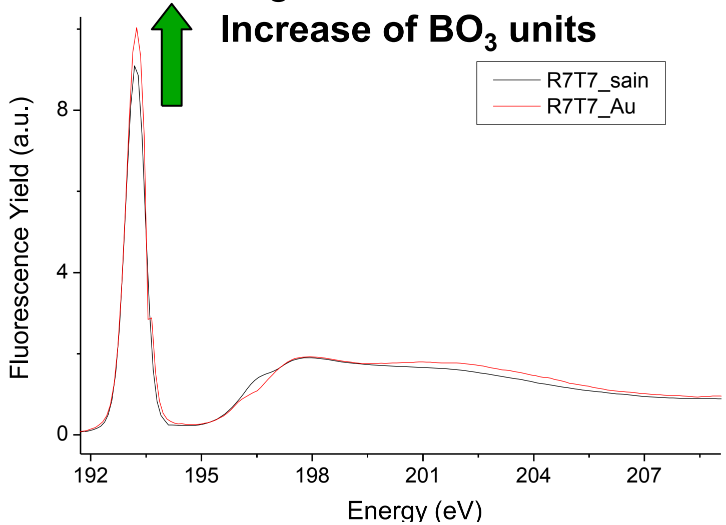
Conclusion :

Partial conversion BO_4 into BO_3 Complex and simplified glasses

BO_3 increase is lower under α decays irradiation

Xanes B K edge: R7T7 irradiated with Au

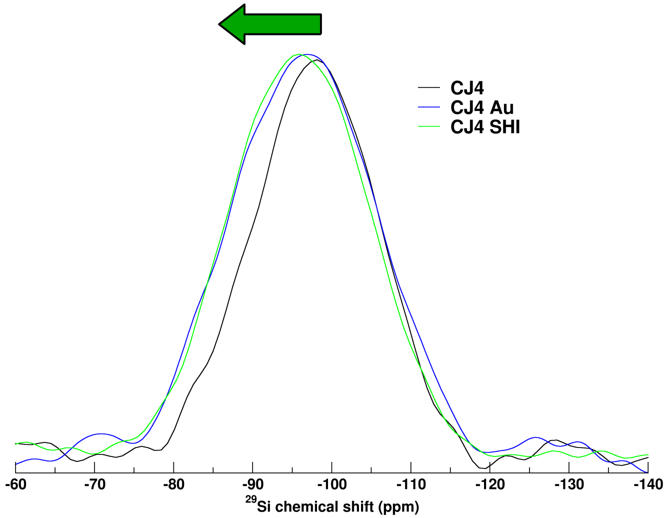
Increase of BO_3 units



G. Bureau, thesis, (2008)

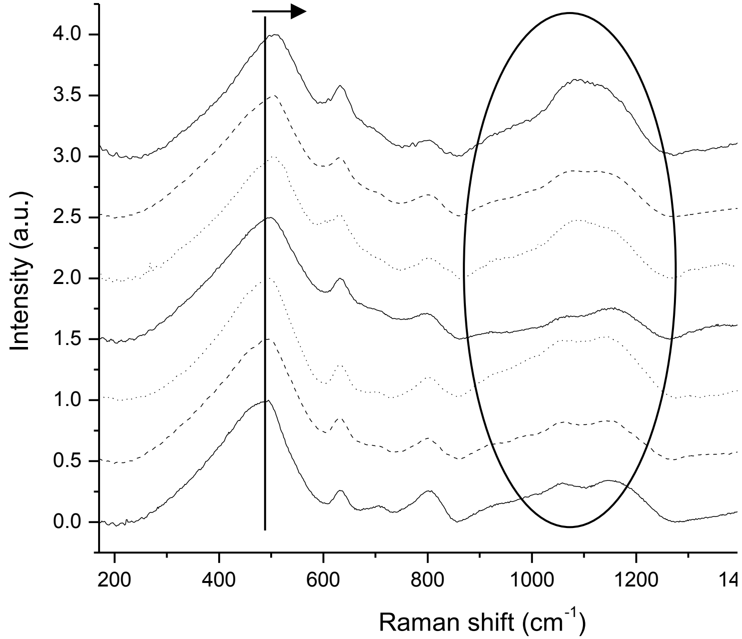
Effect on glass structure : SRO around Si and MRO

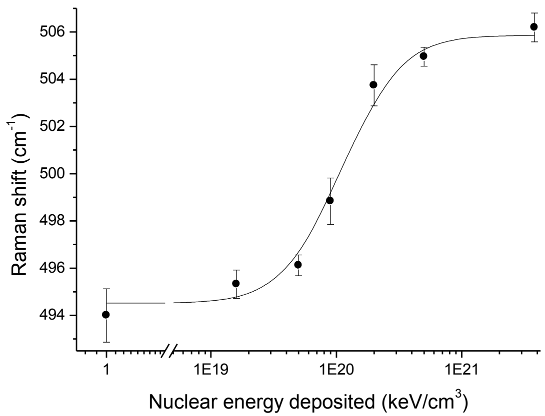
^{29}Si NMR ISG glasses irradiated Au, Xe



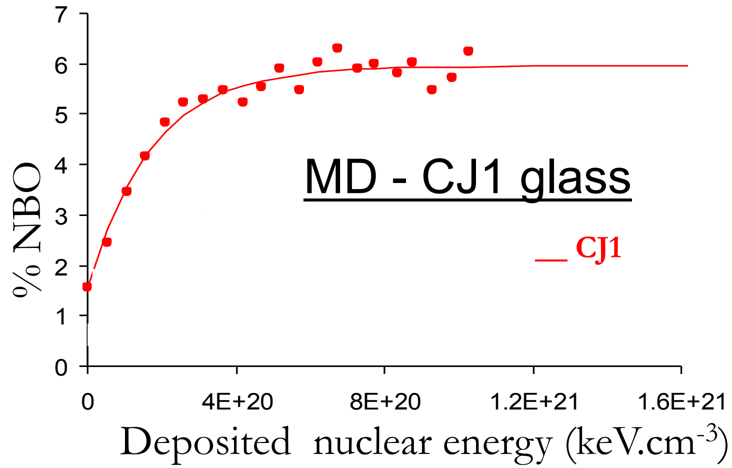
C. Mendoza et al. NIMB 325 (2014) 54-65

Raman spectroscopy on simplified CJ1 glass





J. deBonfils et al. J. Non-Cryst. Solids 356 (2010)



J.-M. Delaye et al, J. Non-Cryst. Solids 357 (2011) 2763

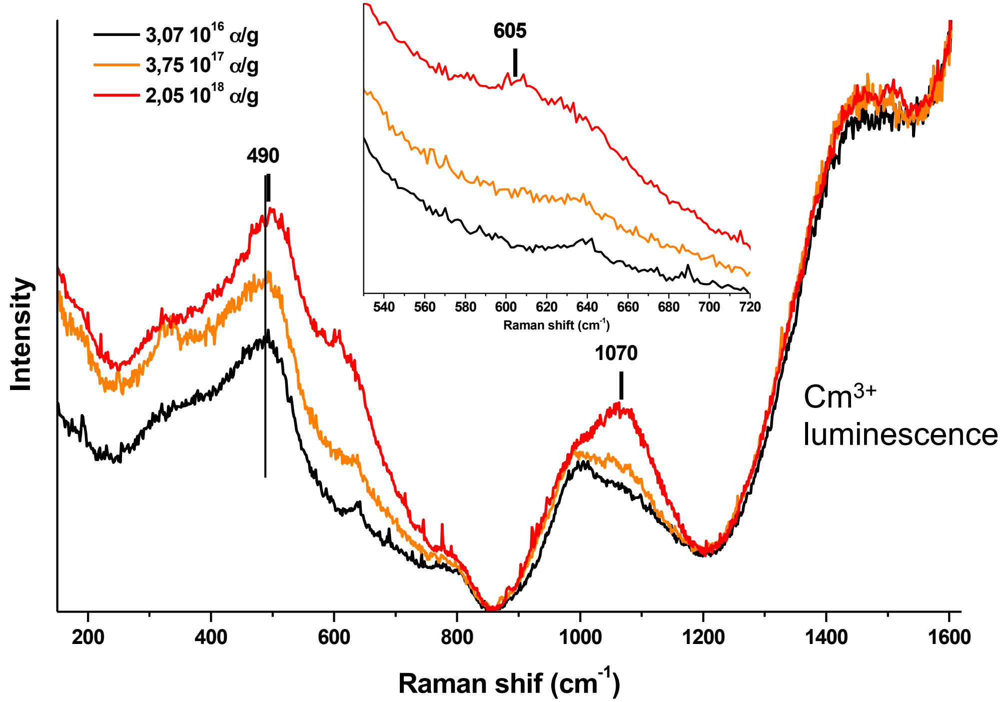
- **Slight depolymerisation of the glassy network of simplified glasses**
- **Shift of the vibration band around 500cm^{-1}**

  → Decrease of the mean angle between silica tetrahedral

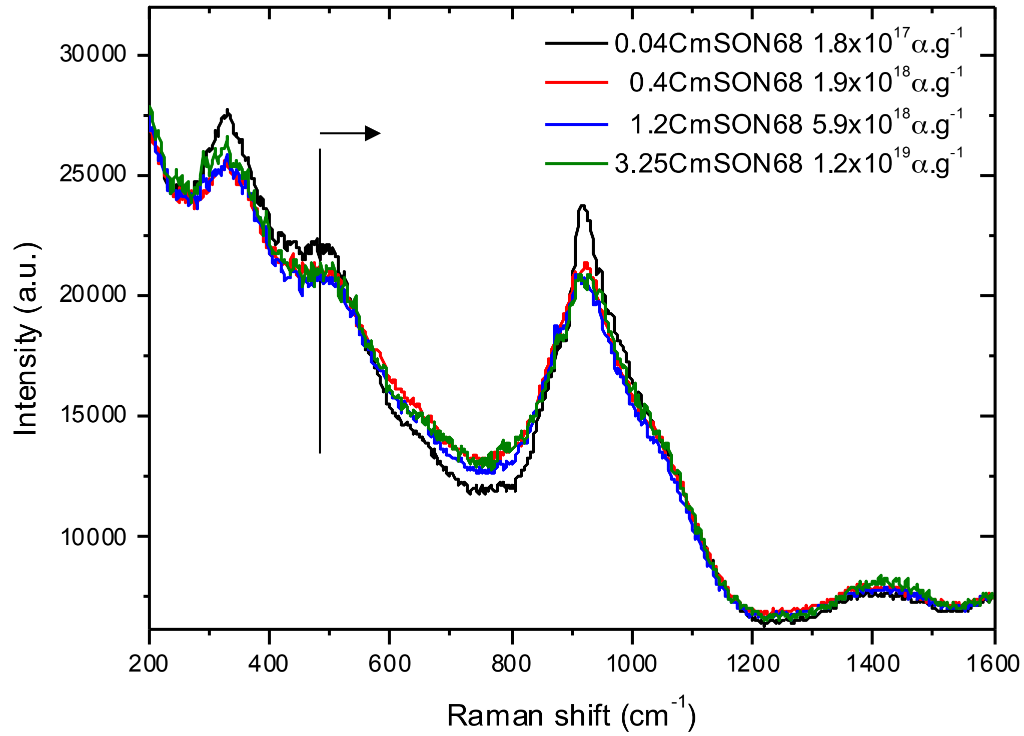
- **Stabilization of the silicon local environment after around 10^{21} keV/cm^3 (~ 4×10^{18} α/g)**

Effect on glass structure : SRO around Si and MRO

Raman spectroscopy on Cm doped ISG and SON68 glass (Atalante, DHA)



C. Mendoza et al. Proc. Chem. 7 (2012) 581



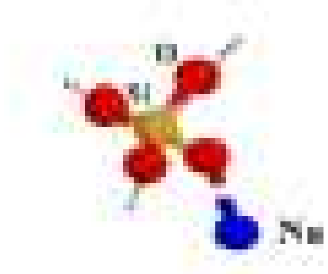
S. Peuget et al. JNM 444 (2014)

- Increase of Q3 contribution in ISG glass : more NBO
- Slight shift of the vibration band around 500cm^{-1}

  Decrease of the mean angle between silica tetrahedra
- New D2 band on ISG Cm doped glass: 3 members silica rings
- Stabilization of the silicon local environment after around 2 x 10^{18} α/g

Modification of the Short Range Order

Increase of trigonal boron, increase of NBO



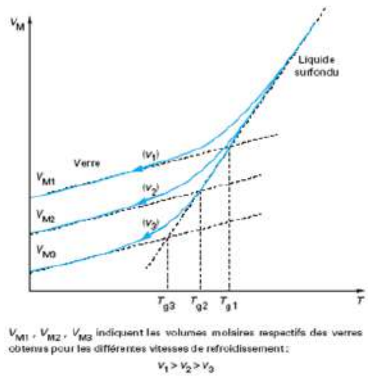
Modification of the Medium Range Order

Ring statistic modification, increase of glass disorder and Si/B mixing

Effects similar to those induced by thermal quenching of a molten glass

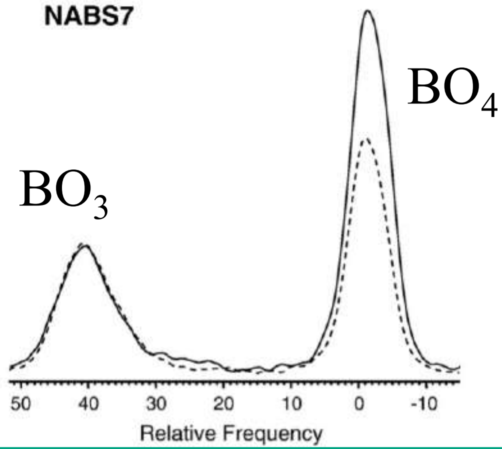
$BO_4 \xrightarrow{T_f \nearrow} BO_3 + NBO$

Decrease of the glass density



^{11}B NMR on quenched and annealed glass

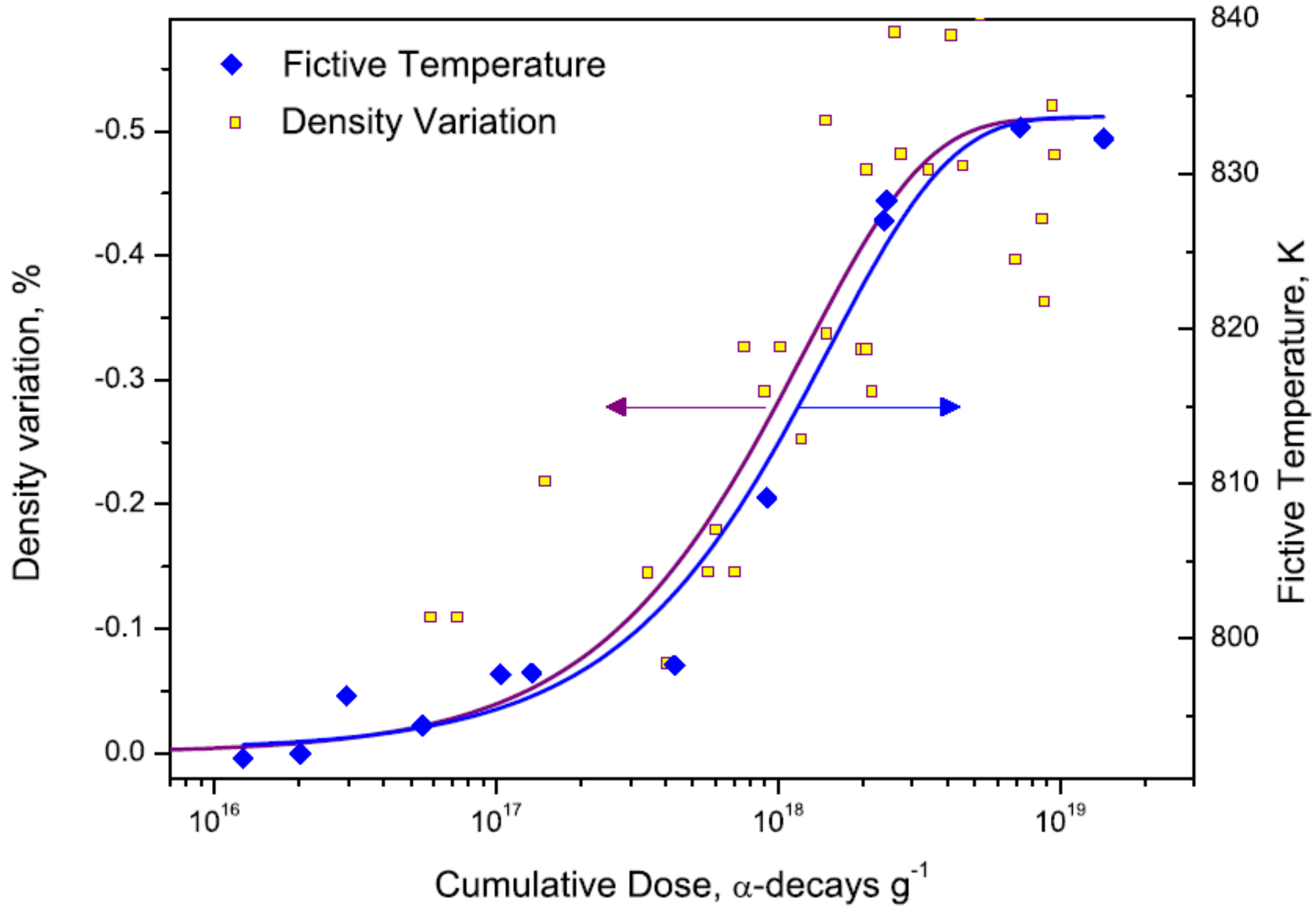
Wu and Stebbins JNCS 356 (2010)



3- Effects on the glass structure? Fictive temperature

E Maugeri et al, J. Am. Ceram. Soc. 95 (2012) 2869

DSC on ^{244}Cm doped SON68 glass (ITU, actinet-i3 project)

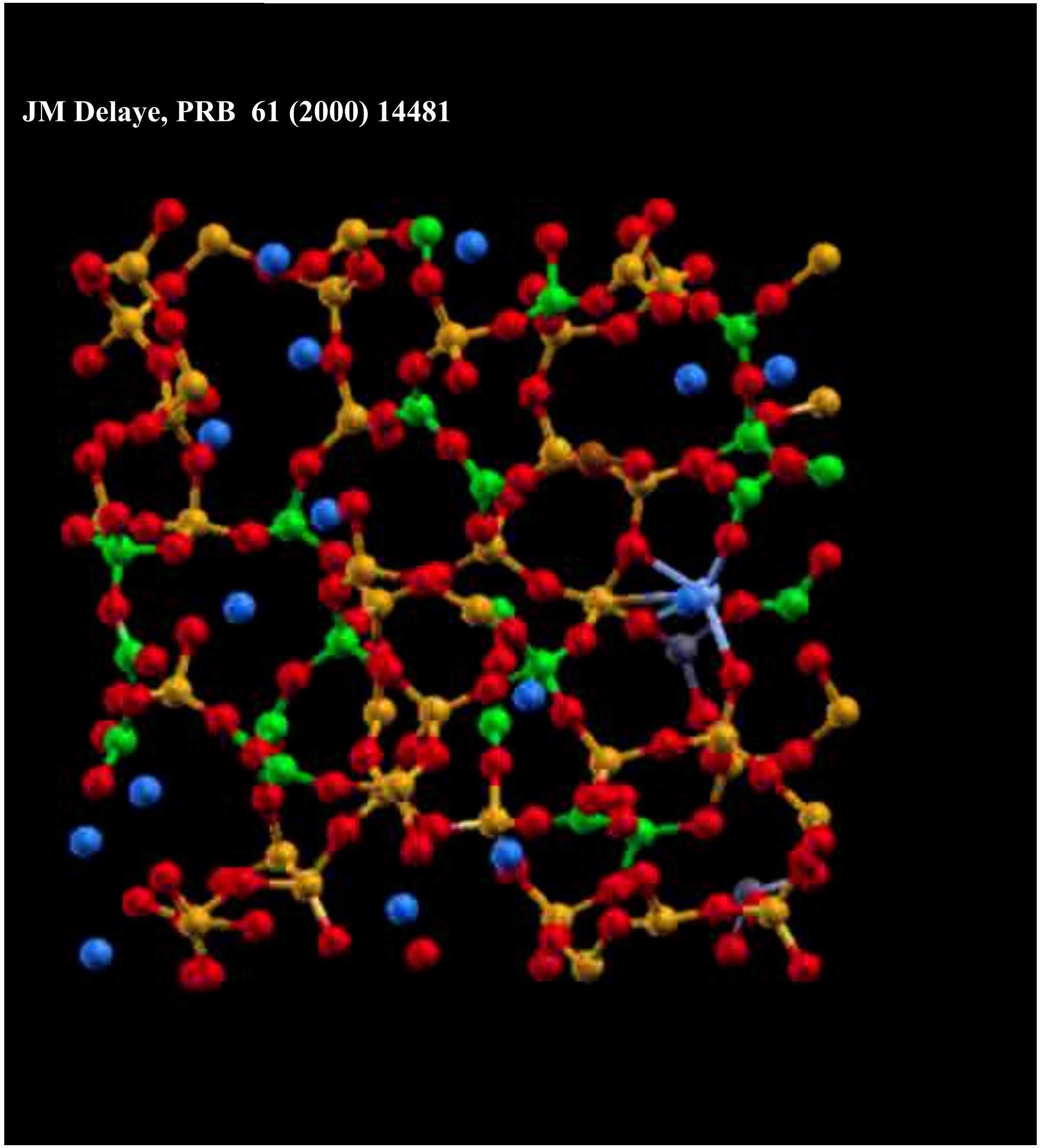


Increase of the glass fictive temperature with alpha decay dose

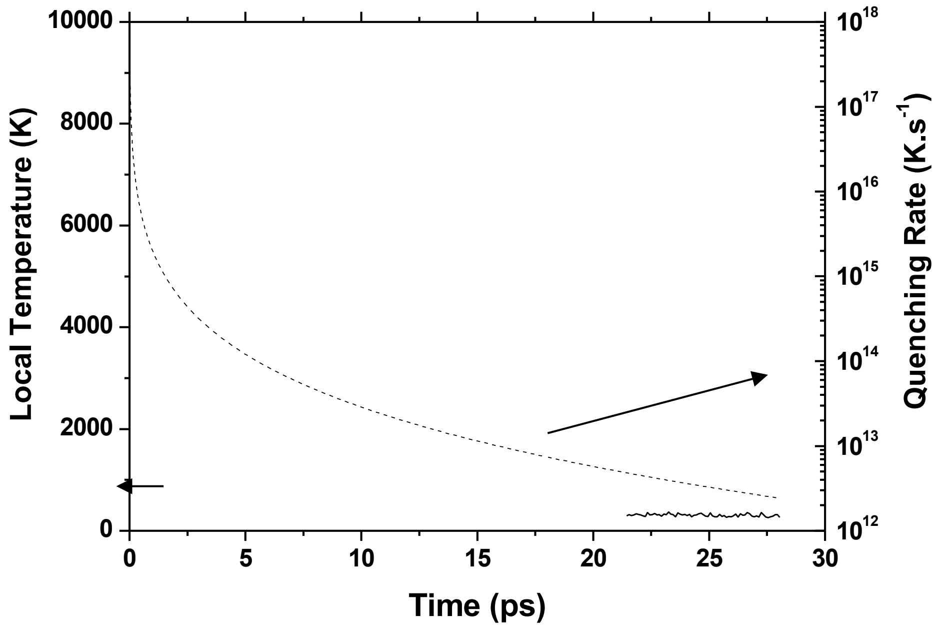
Formation a new structure similar to a fast quenched glass
New metastable phase induced by irradiation

3- Ballistic damage : why the glass evolve?

What happen in the displacement cascade induced by a recoil nuclei?



1. Balistic phase
2. Thermal phase



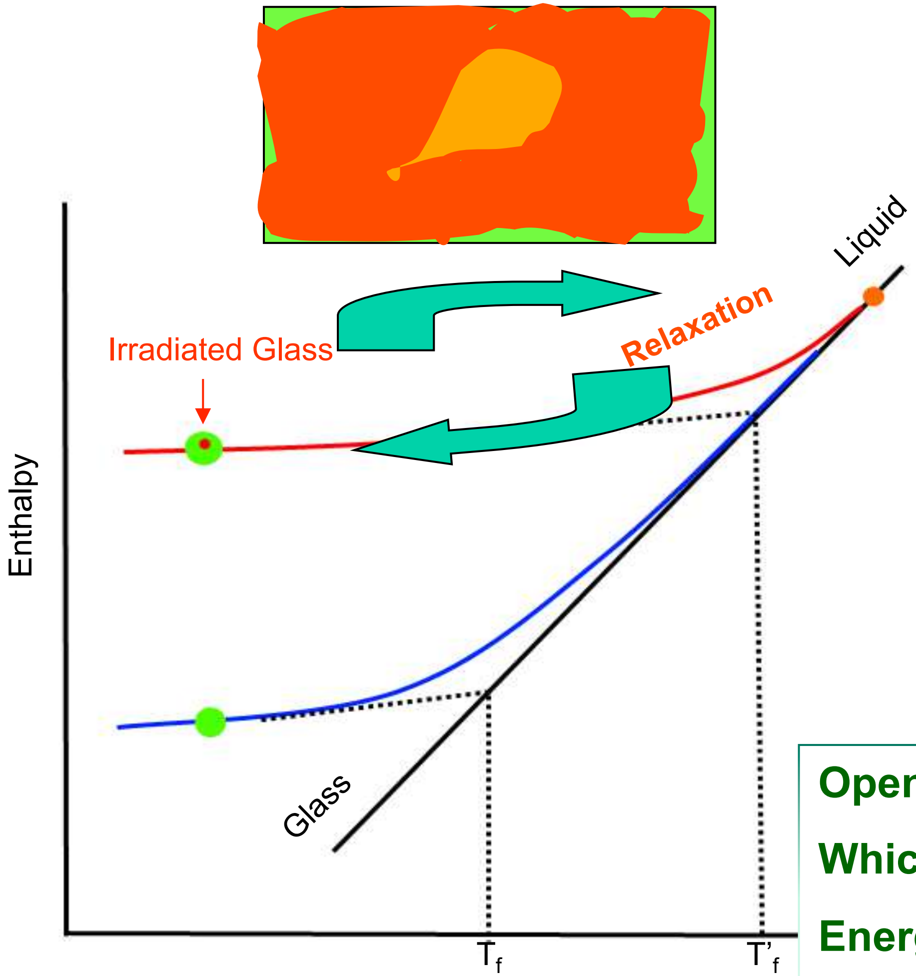
Golden = Si
Green = B
Blue = Na
Red = O

Very high quenching rate of the disorder state induced by the displacement cascade

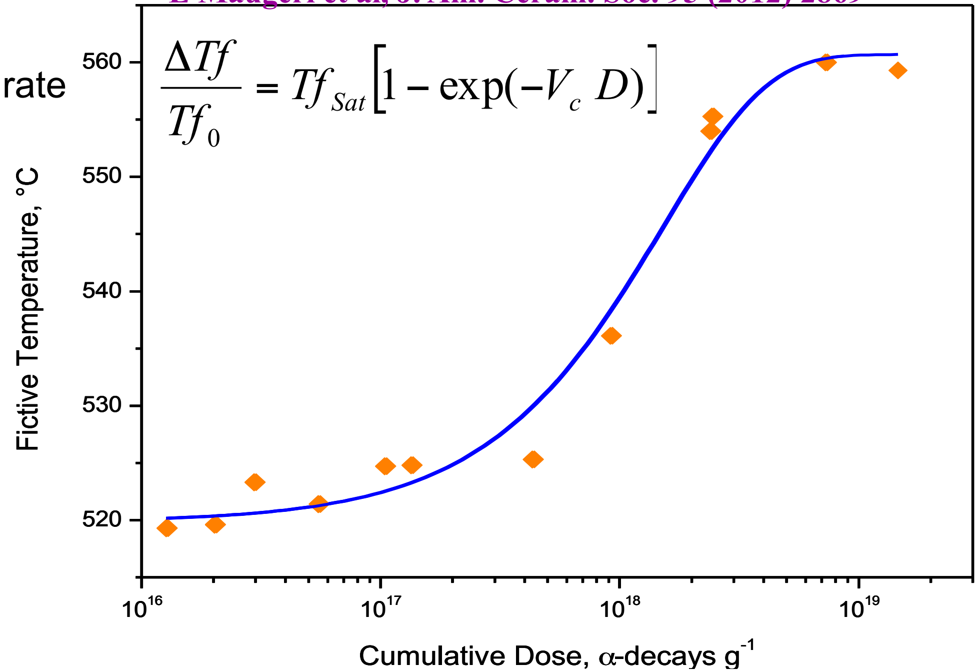
3- Understanding of glass behavior under alpha decays

1. Balistic step : disordered state
2. Relaxation phase : very important quenching rate

Irradiated zone has a higher fictive temperature



E Maugeri et al, J. Am. Ceram. Soc. 95 (2012) 2869



Model of accumulation of ballistic disordering fast quenching events: supervitrification

Stabilization of a new glass structure when all the volume has been damaged once time

Open questions:

Which step control the irradiated state?

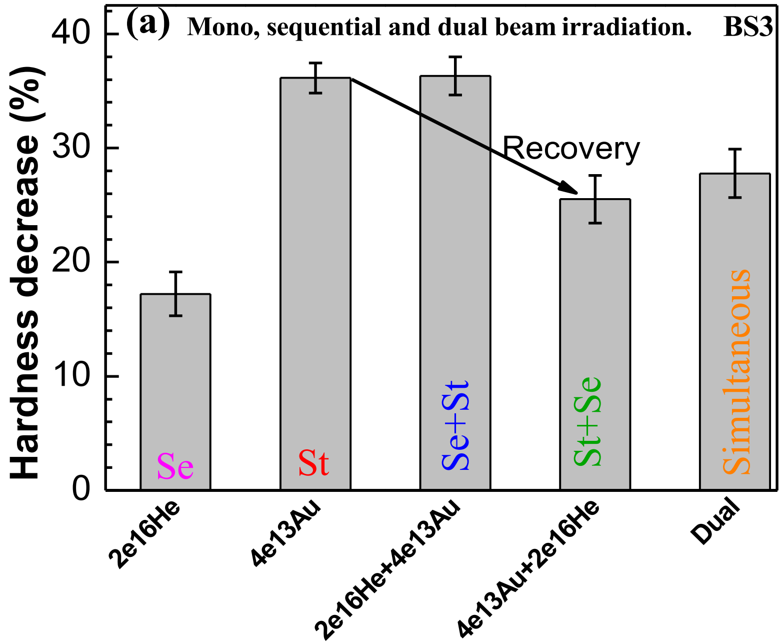
Energy deposition step, quenching step?

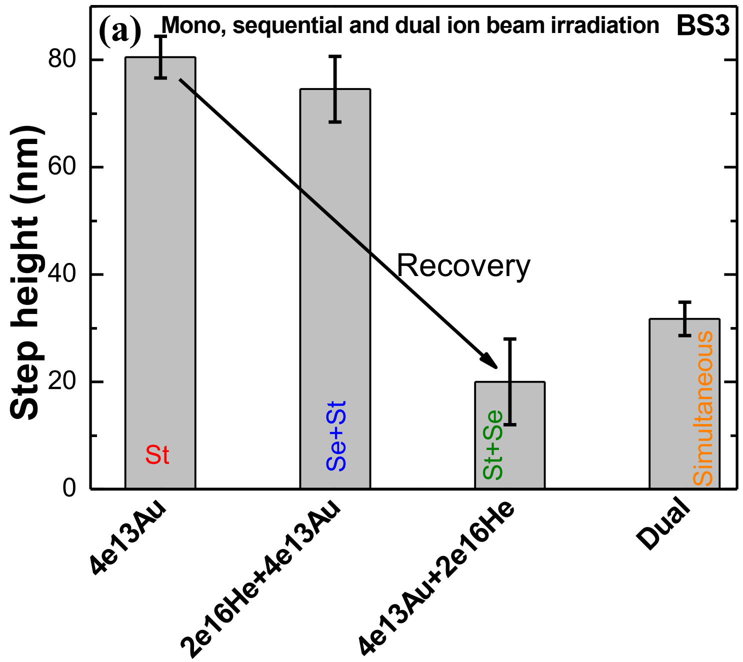
Why some property variations are lower in α doped glass

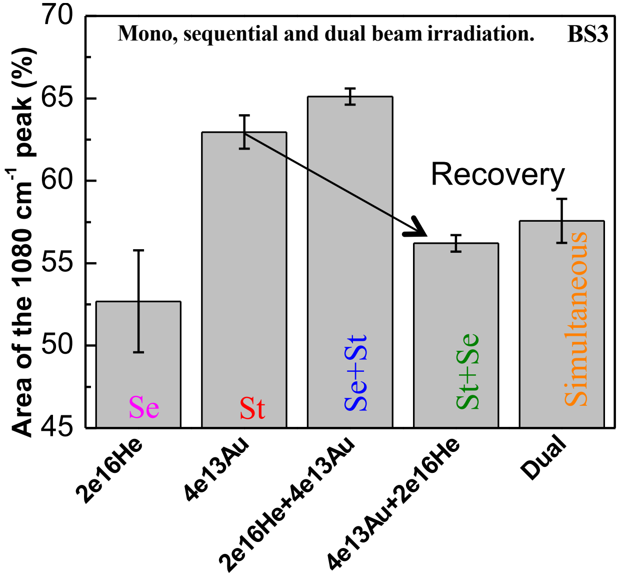
Which step control the irradiated glassy state?

Recent results using double beam irradiations

A.H. Mir et al, accepted in Eur. Phys. Lett.

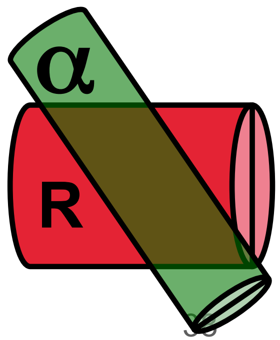






Role of both nuclear and electronic stopping powers

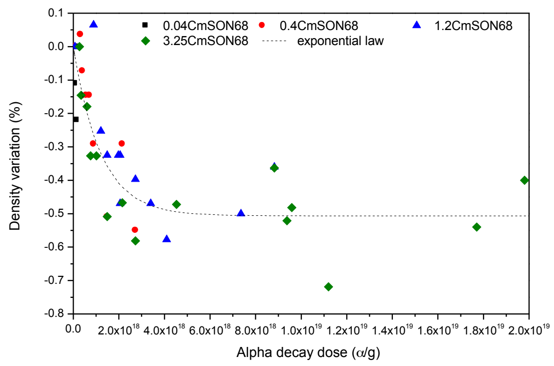
- Heavy ions: main damage (supervitrification)
- Alpha particle: recovery effect due to electronic energy loss



Explain the lower property variation observed on α doped glass compared to heavy ions irradiated glass

Conclusion on alpha decays effects

- Slight modification of density and mechanical properties
- Glass is still homogeneous (SEM and TEM scale)
- No effect on initial alteration rate
- Modification of glass Short Range Order (boron coordination, NBO …)
- Modification of Medium Range Order (ring statistic, angle distribution)
- No effect of accelerating the time scale



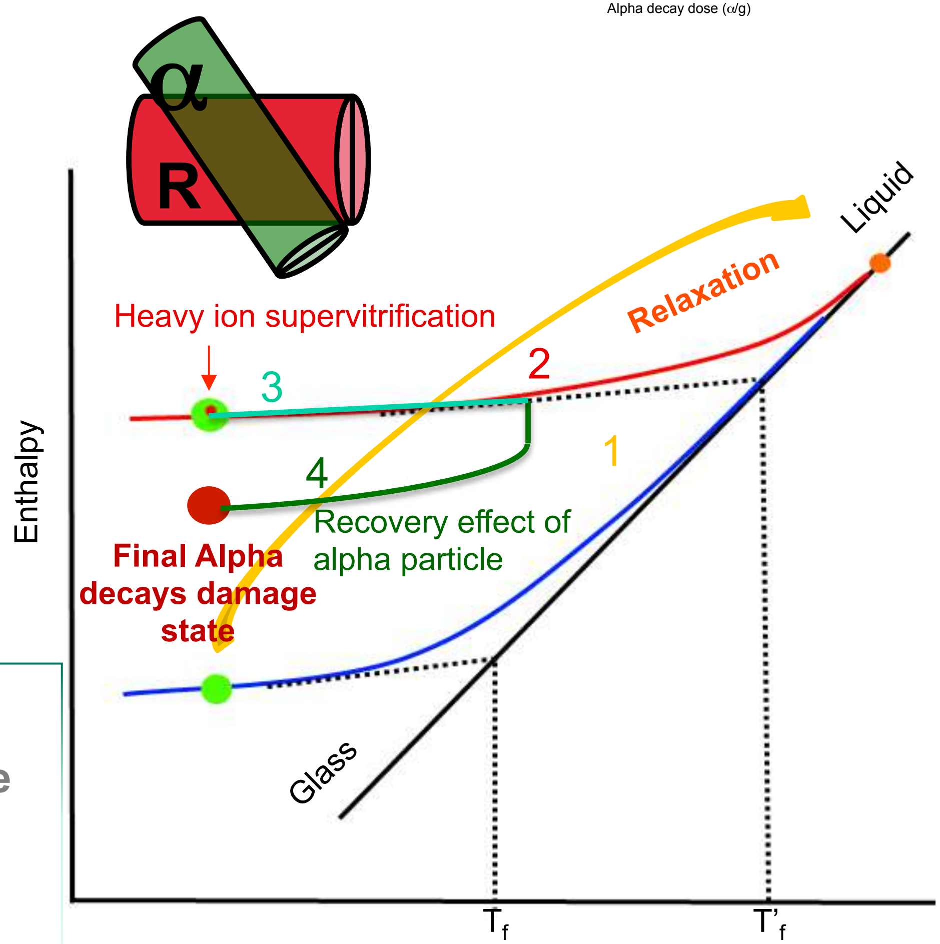
Modifications observed in the first $4x10^{18}\alpha/g$ according to a direct impact model

Saturation when all the glass has been damaged by recoil nuclei events and alpha particles

Recoil nuclei : supervitrification of the glass (1&2)

Alpha particles : partial recovery of the damage (3&4)

No limitation of alpha decays accumulation?



Prospects :

Effects of alpha decay dose on long term alteration rate

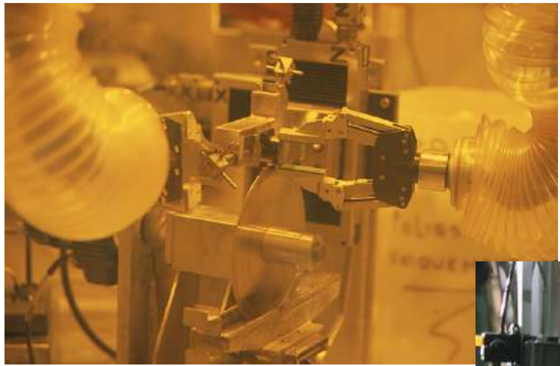
Coupling alpha and beta decays and thermal history

Is helium generation a problem?

Thank you for your attention !!!

Special Thanks to

DHA - Atalante



DE LA RECHERCHE À L'INDUSTRIE

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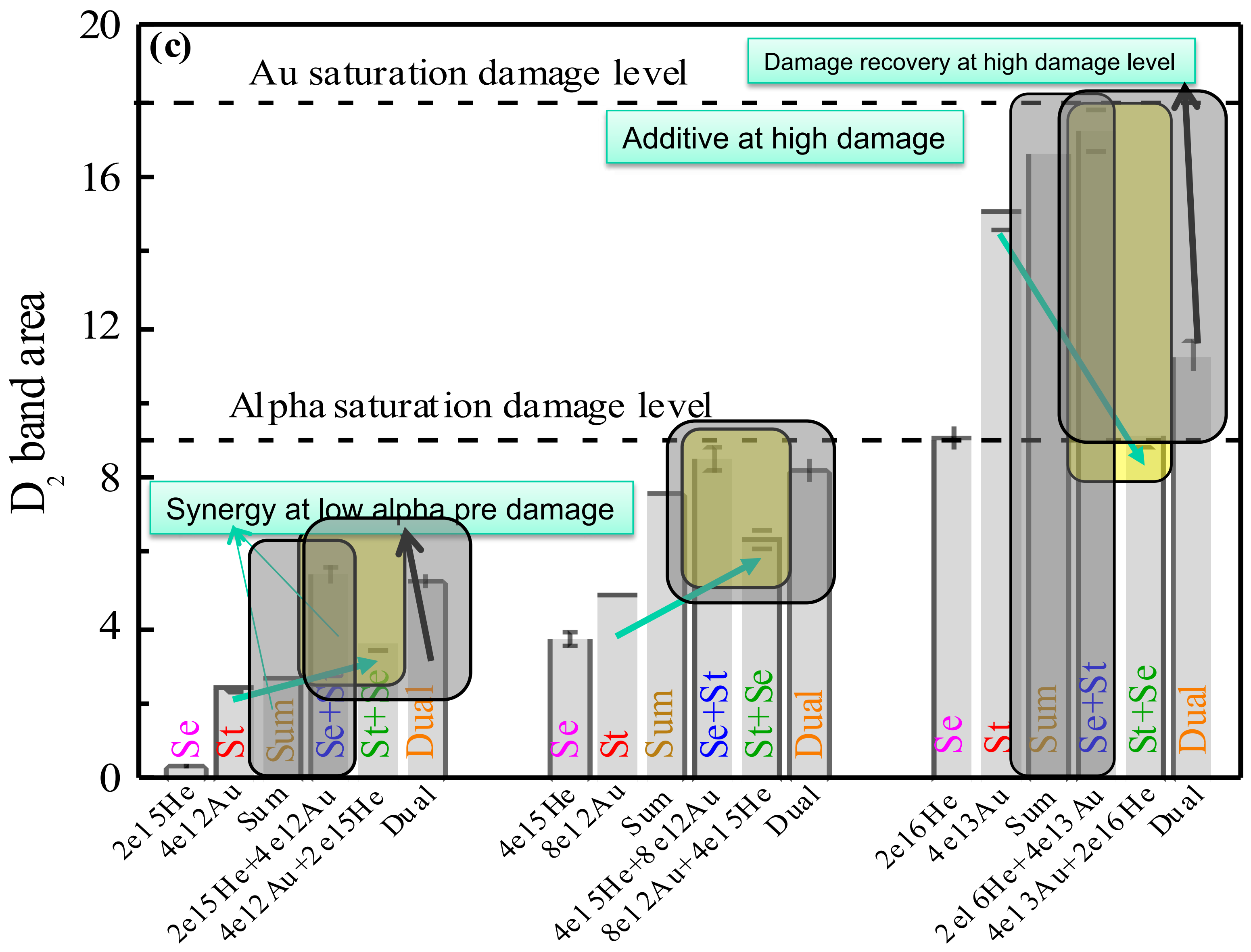
ACTINET 3 – Integrated Infrastructure Initiative for Actinide Science

EURACT-nmr

LCV Joint Vitrification Lab – cea AREVA

AREVA

Damage scenarios: Comparison at different damage levels



Se = α

St = Au

Se +St = α + Au

St+Se = Au + α

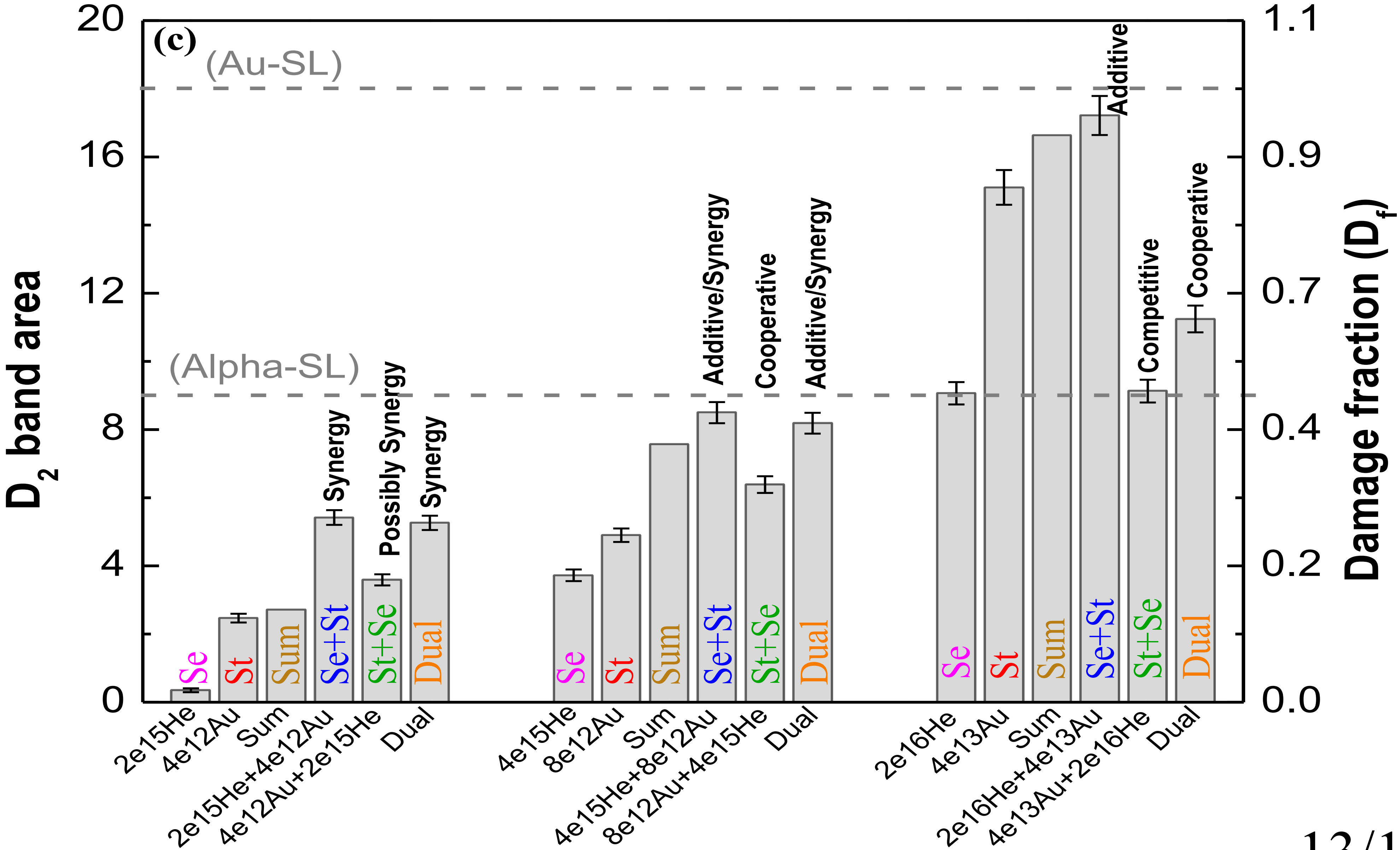
Dual = Simultaneous (Se & St)

Sum??

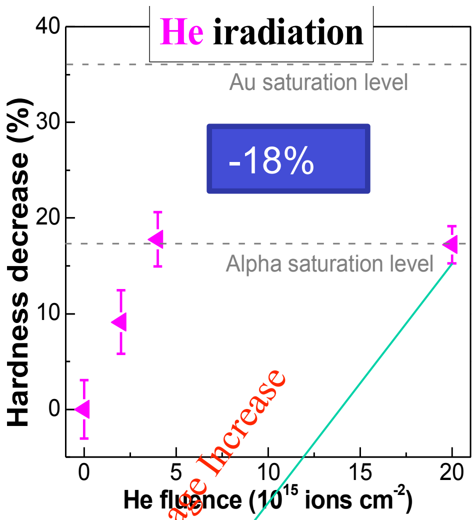
Dual

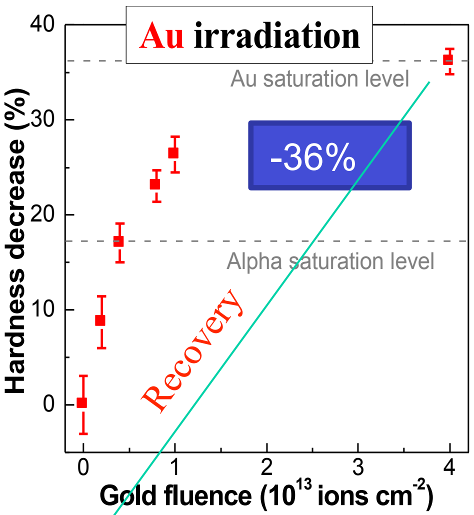
12/15

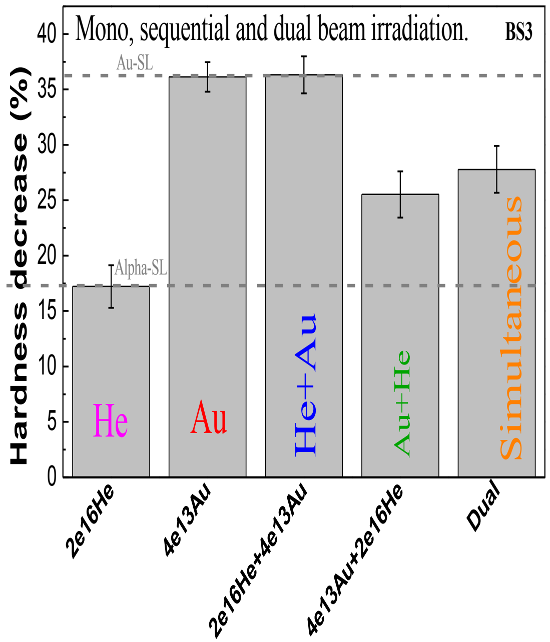
Different Damage processes

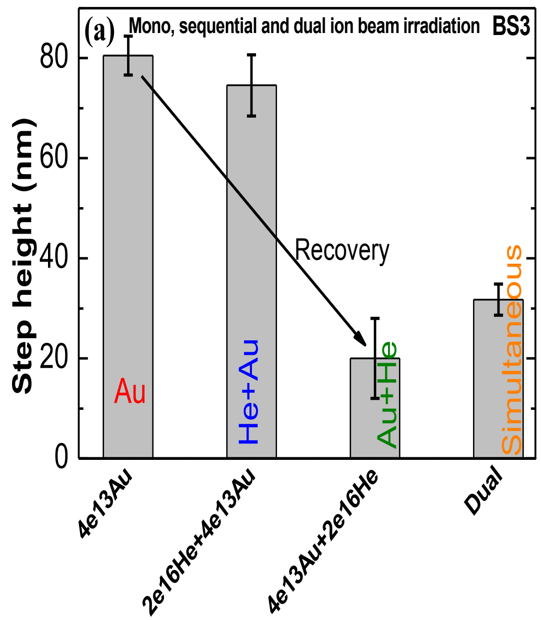


Double ion beam irradiation induced changes

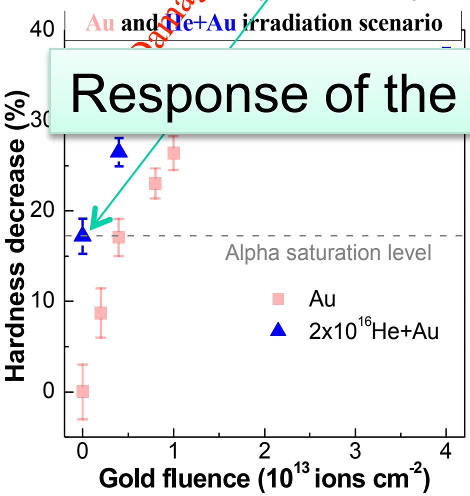


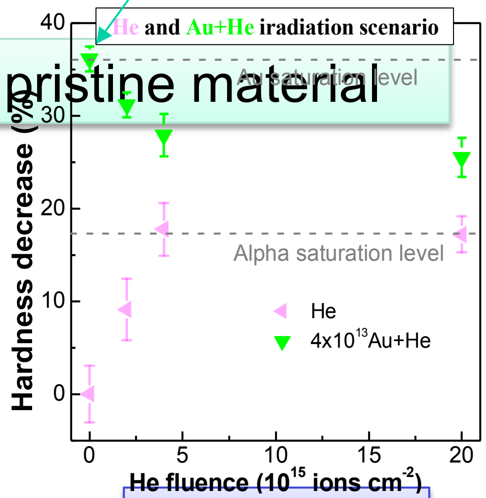






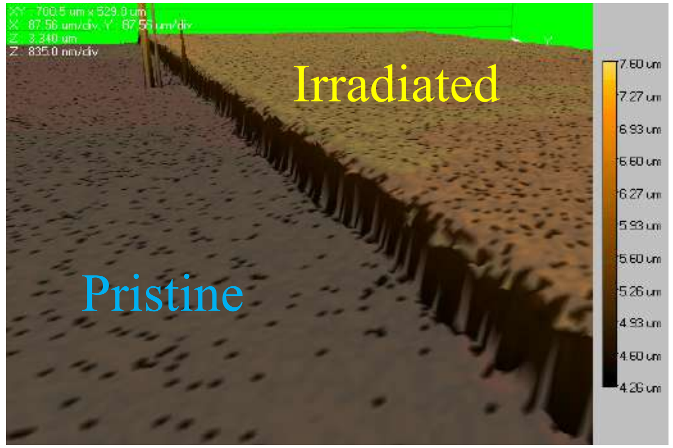
Response of the pristine material





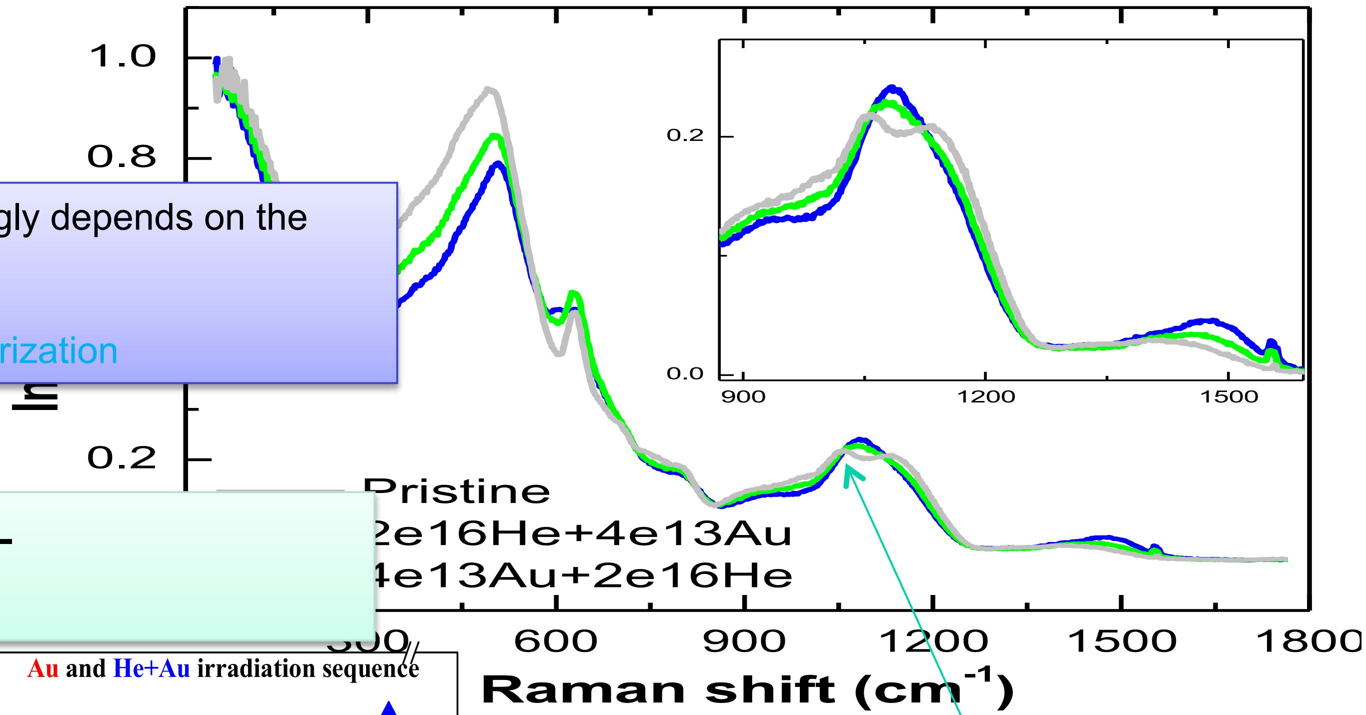
He+Au~Au

Au+He~He



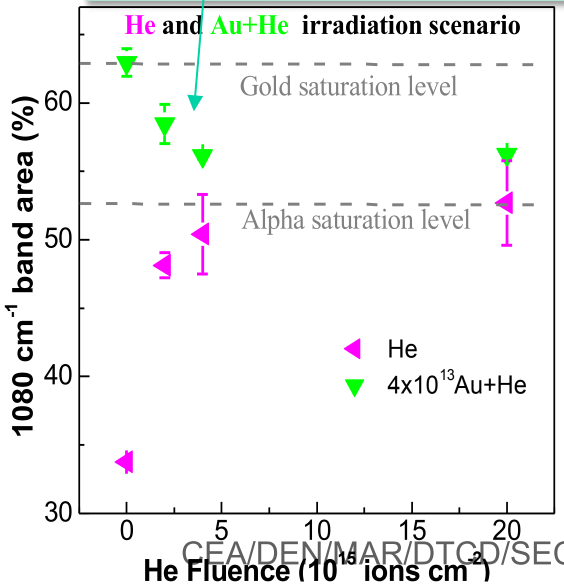
Response of Pre-damaged material

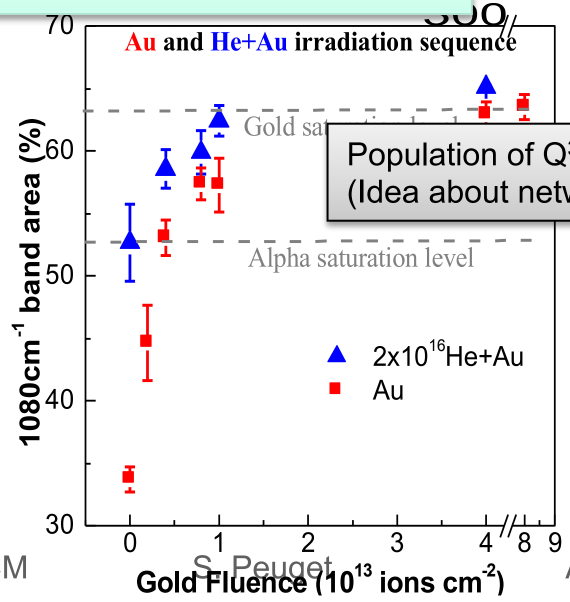
Double ion beam irradiation induced changes



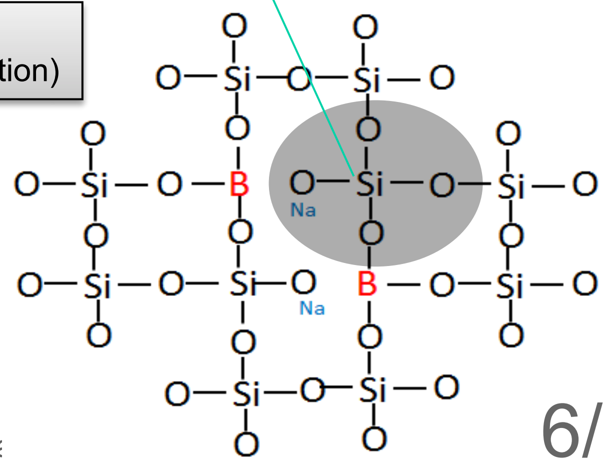
Response to alpha irradiation strongly depends on the pre-existing state.
Pristine: Depolymerization.
Au Pre-irradiated: Partial re-polymerization

Alpha induced partial re-polymerization



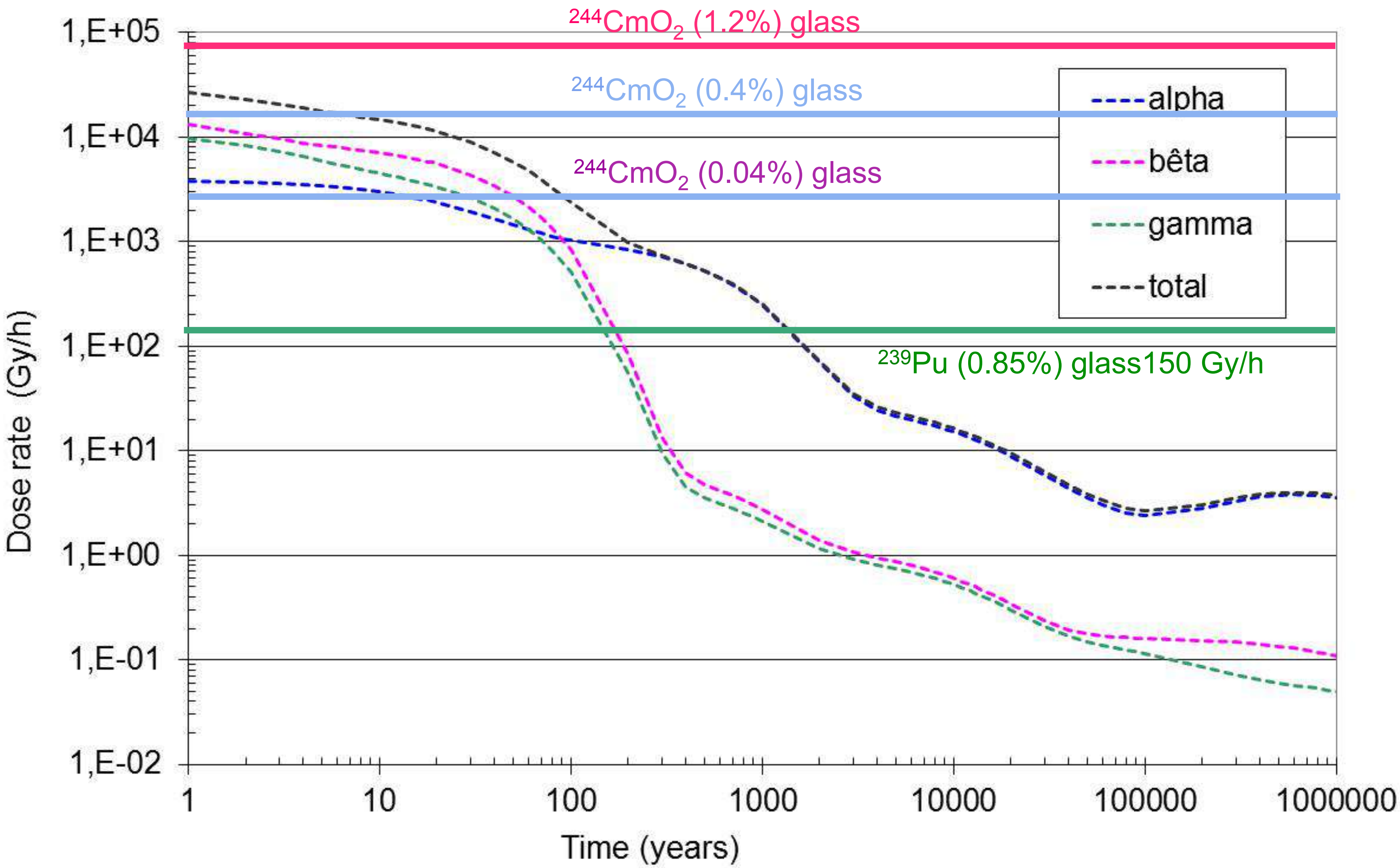


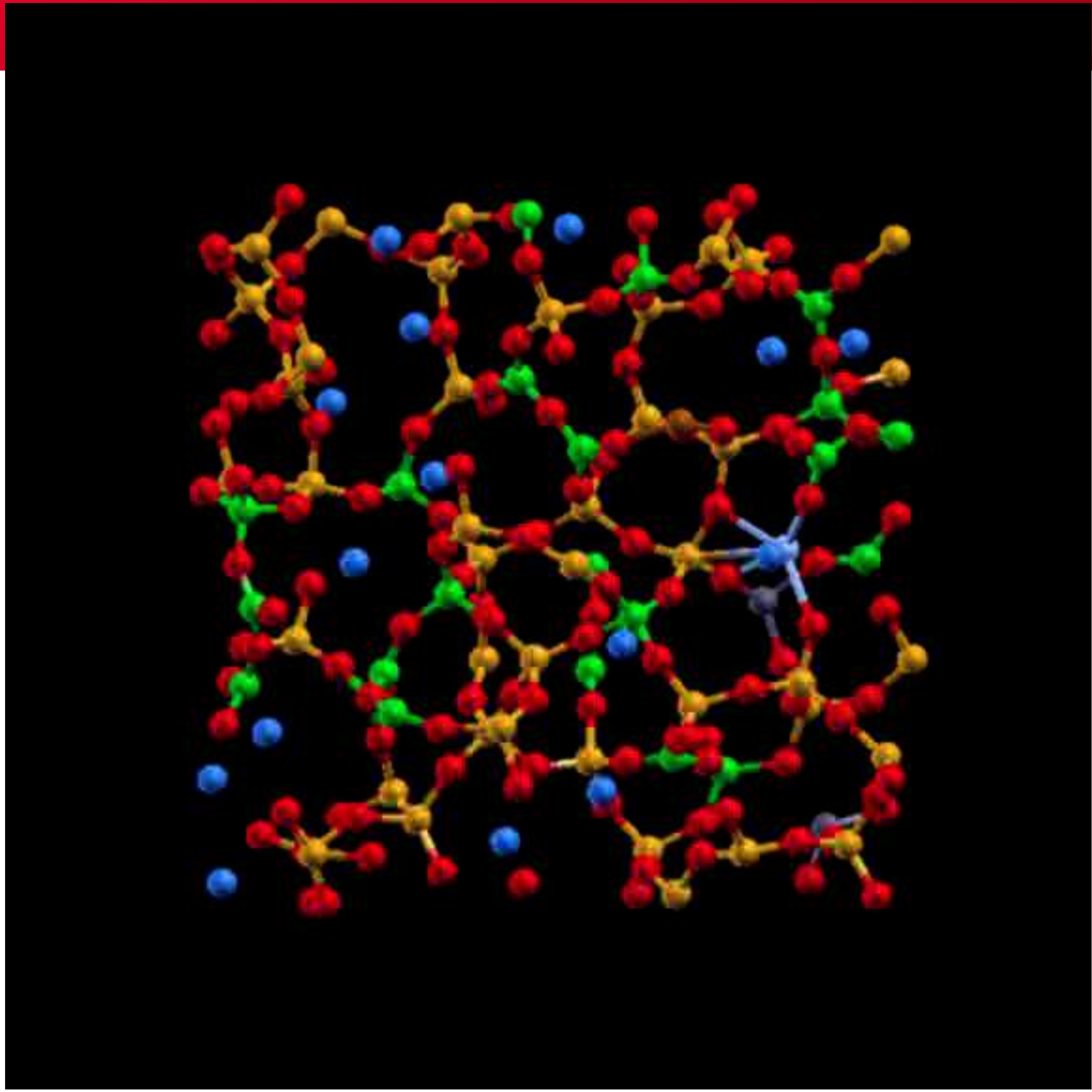
Population of Q^3 units
(Idea about network Polymerization)



Glass LTB: Leaching, coupling with irradiation

Simulation of alpha dose rate





Effects on the glass structure: irradiation / quenching rate

- **Modification of the local order**
  - Partial conversion of BO_4 in BO_3
  - Increase of NBO on silicon atoms

$BO_4 \longrightarrow BO_3 + NBO$

Irradiation

Temperature

Irradiation is similar to a very high quenching rate effect

- **Modification of the medium range order**

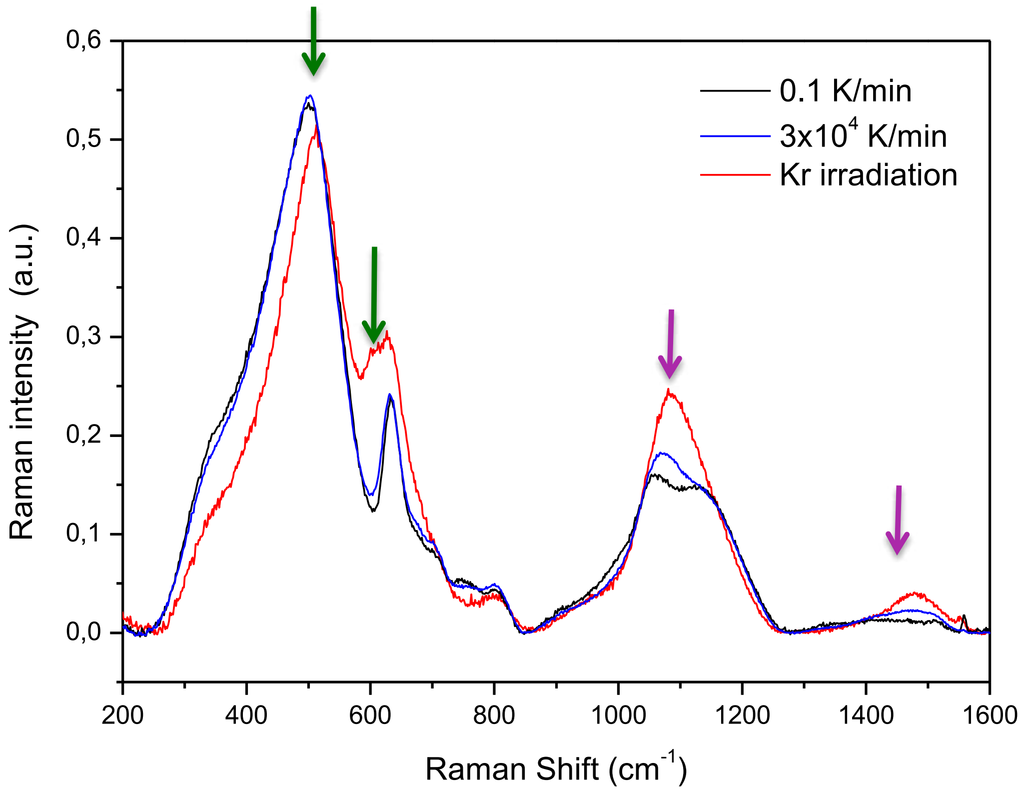
  Decrease of Si-O-Si angle

  Increase of 3 member's silica rings

  Increase of the glass disorder

Specific to irradiation effect

Comparison of irradiation and quenching rate



S. Peuget et al. JNCS 378(2013)

Glass LTB: Effect of beta decay

Oxide glasses are sensitive to electronic excitation

Optical materials:

Increase of LASER power and decrease of pulse duration

Production of point defects (fibers, lens)

a-SiO_2, Er-, Yb - doped glasses

Improving Glass properties with respect to ionisation phenomena

Sm – doped glasses, Ti – doped glasses, Yb – doped glasses

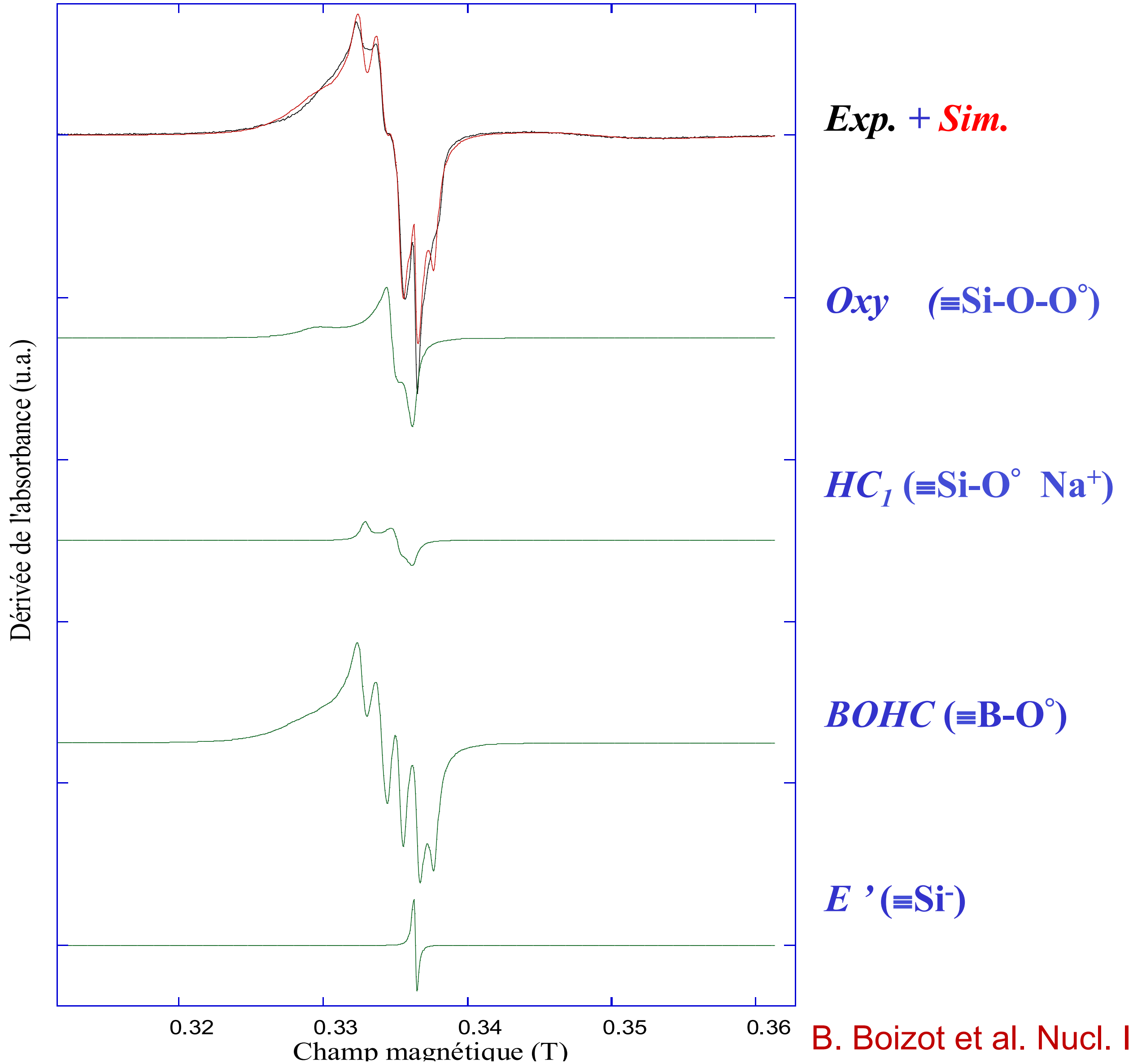
Approach for studiyng irradiation effects in the matrix of nuclear glass

- Simulation of beta decay by electron irradiation
- External irradiation with electrons of nuclear glasses

NEC pelletron accelerator

- Study of structure after irradiation by NMR, EPR, Raman and Photoluminescence
  Mechanisms occuring during electronic excitation in glasses
- Complexification with a perturbation approach
  Mixed alkali effect (K/Na, Li/Na, …)
  Doping wih TM (Fe, Cr) and RE (Sm^{3+}, Gd^{3+}) doped borosilicate glasses

Defects production in simplified oxide glasses (EPR)



Ionization produces Excitons

↓

[hole centers] >> [electron centers]

↓

Accumulation of ponctual defects

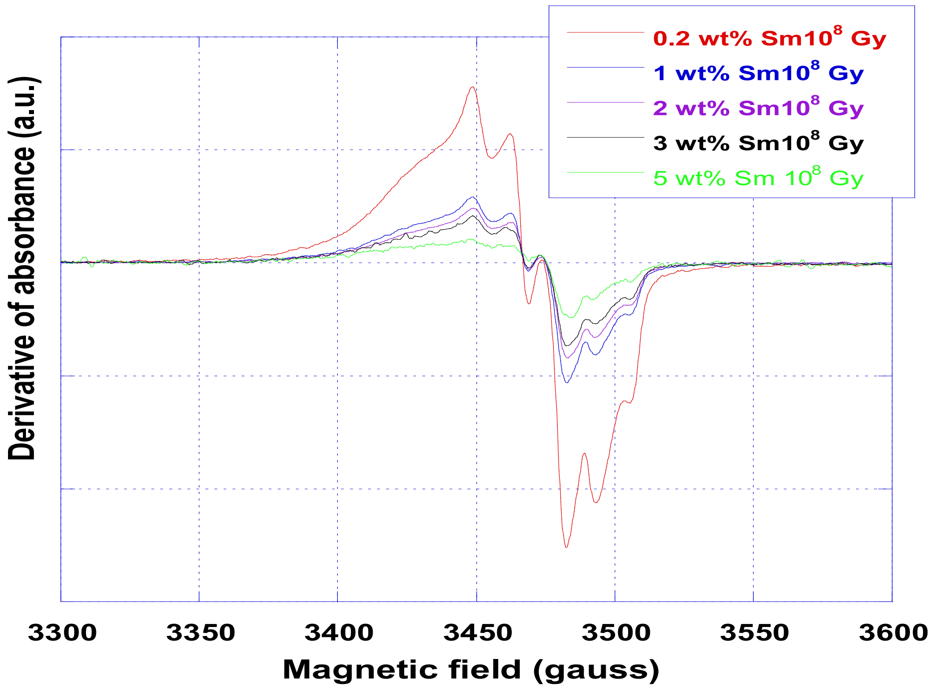
↓

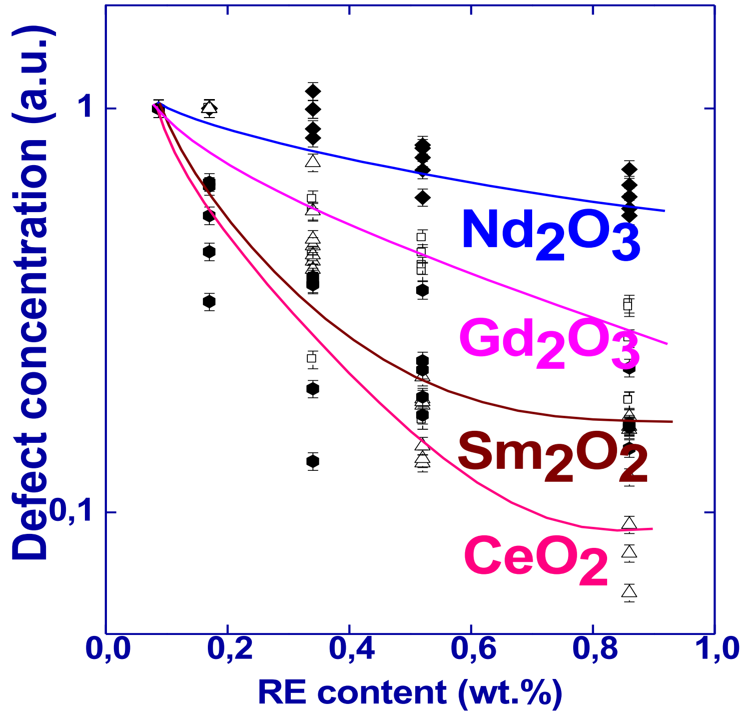
Structural evolution of the glass

B. Boizot et al. Nucl. Instr. and Meth. in Phys. Res. B 141 (1998) 580.

Glass LTB: Effect of beta decay

Effect of incorporation of lanthanides





Strong influence of doping on the defects production

- *Process consuming exciton production*
- *Trapping depends on the nature of doping ion*

E. Malchukova et al, J. of Non_Cryst. Solids 353 (2007) 2397.

Glass LTB: Effect of beta decay

Effect of incorporation of transition metals (Fe, Cr)

Ionizing radiation

Thèse de F Olivier (Ecole Polytechnique) 2006
N Ollier et al, JNCS 323 (2003) 200

↓

Exciton creation (e^-/h^o) but interaction with the transition elements

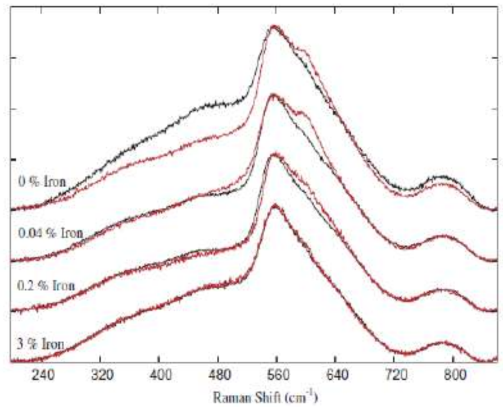
Equilibrium:

$Fe^{3+} + e^- \longrightarrow Fe^{2+}$

$Fe^{2+} + h^o \longrightarrow Fe^{3+}$

↓

Decrease or vanishing of structural evolution



The glass complexity (transition metals, lanthanides, mixed alkalines …) increases the glass resistance to ionizing radiation

No significant evolution of SON68 glass properties after irradiation with electrons