



# Irradiation and thermal processing of silicates in the laboratory: Insights in extraterrestrial grains properties

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# Outline of the talk

- Context and motivations of the study
  - Dust life-cycle
- Methodology
  - Ionic irradiations
  - Thermal annealing: Experimental protocol
- Some results
- Astrophysical implications
- Summary

# Context and motivations

**Dust:** Responsible of starlight extinction in the lines sight towards distant stars.

## Why study dust?

**Dust** ~ 1% of the total mass in protoplanetary discs.

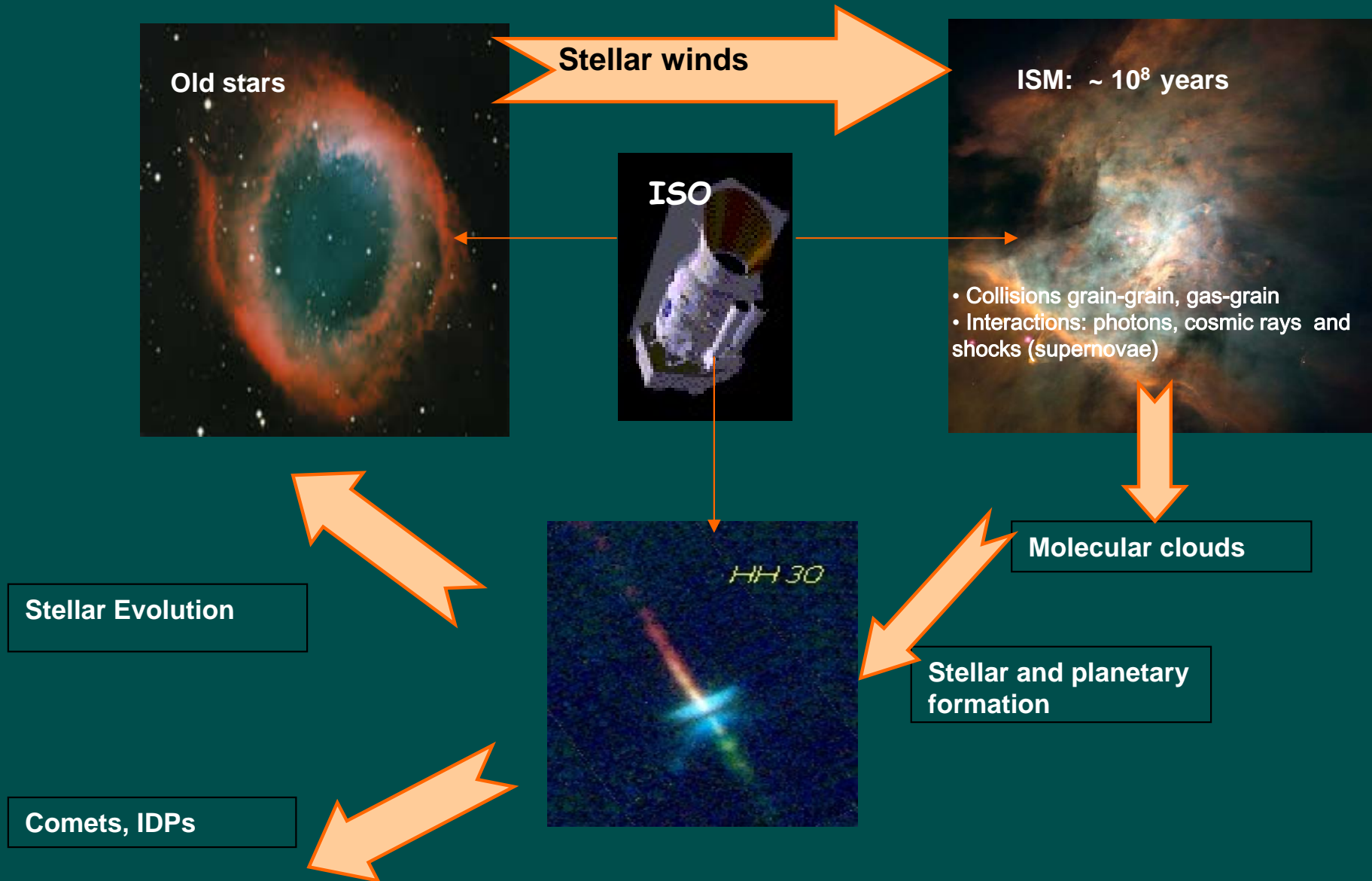
- Represents the building blocks of planets.
- Probes the physical conditions in the different environments

## How is dust observed ?

**Only by spectroscopy: UV, Visible, UV.**

- UV: chemical composition (Carbon)
- Visible: size (modélisation of the extinction curves)
- IR: vibrational: chemical and structural composition  
(crystal/amorphous)
- far IR: lattice vibrations

# Dust life cycle



# Silicate dust

## ➤ Definitions of Silicate:

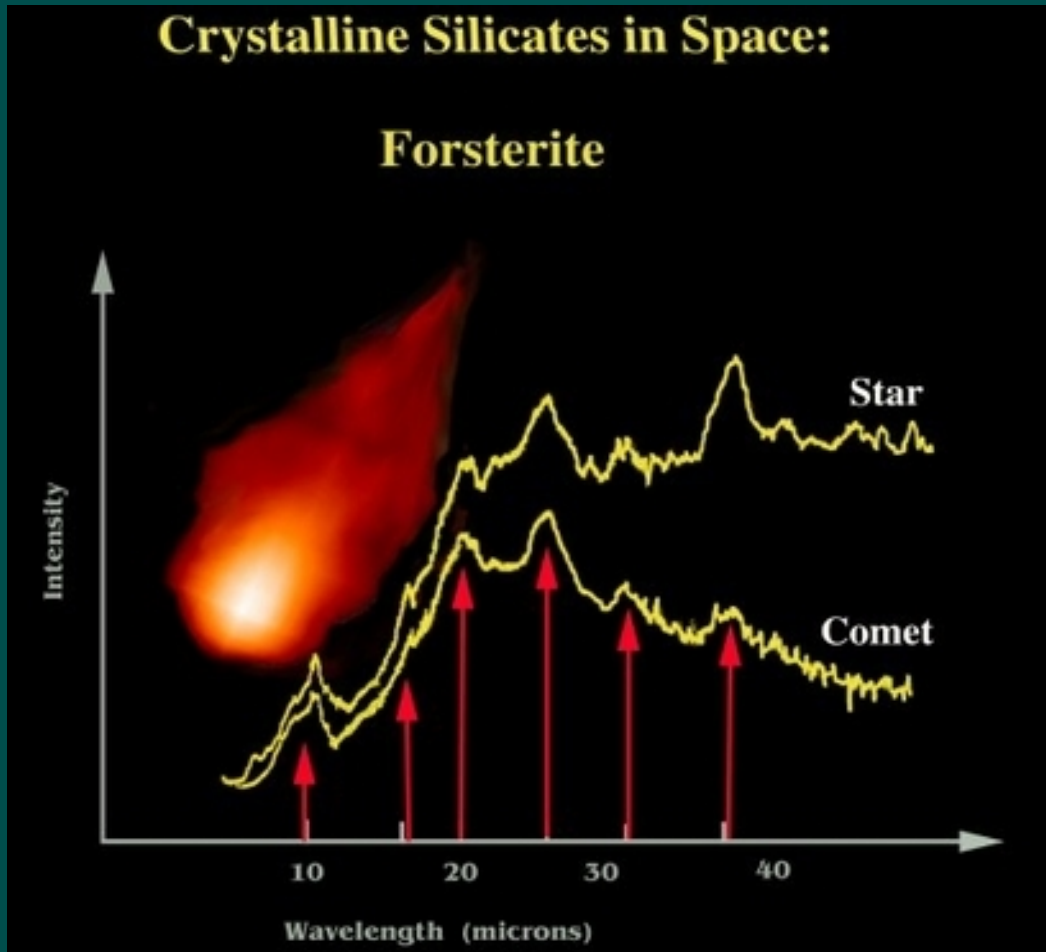
- mineralogical = solid with base unit of Si-O bonds
- astronomical = presence of Si-O bonds (~ 10 and 20  $\mu\text{m}$ ).

## ➤ Dominant forms of Silicates:

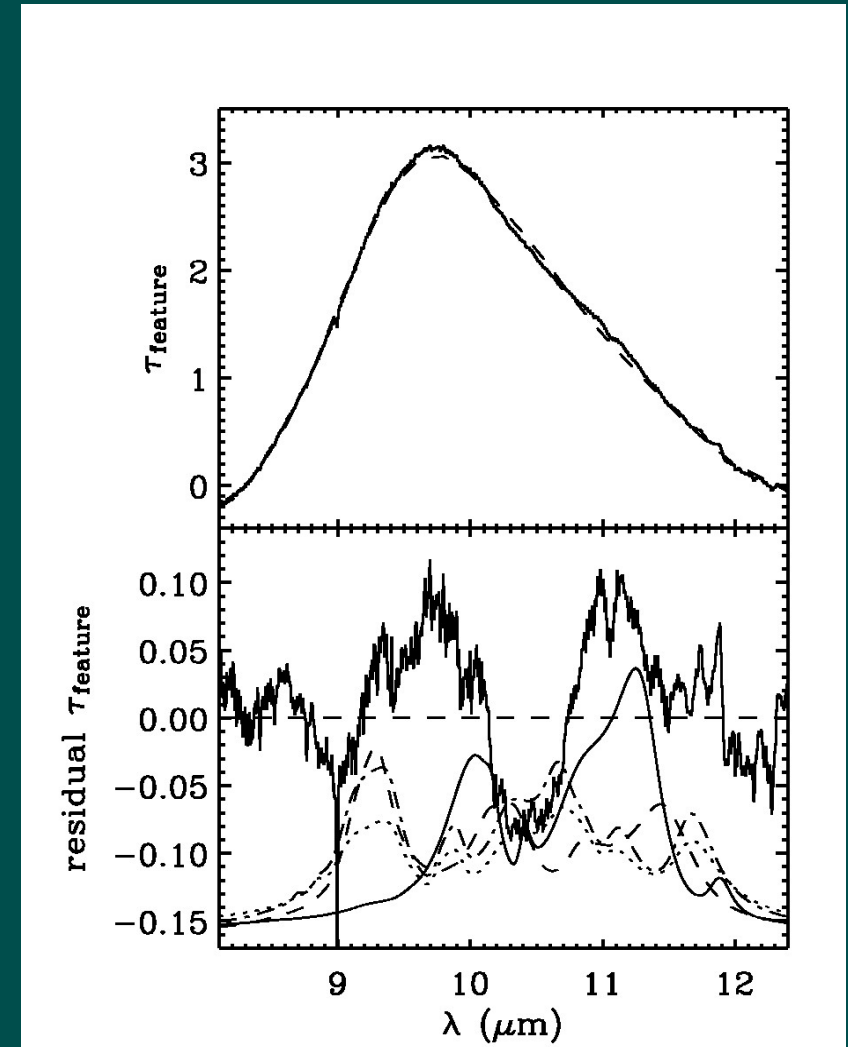
- Olivine:  $\text{Mg}_{2x}\text{Fe}_{2-2x}\text{SiO}_4$
- Forsterite:  $\text{Mg}_2\text{SiO}_4$
- Pyroxene:  $\text{Mg}_x\text{Fe}_{1-x}\text{SiO}_3$
- Enstatite:  $\text{MgSiO}_3$

# Some ISO spectra... The crystalline revolution

Until 90's dust was always thought to be amorphous in space.  
But..



Date: 02 Feb 2000 , Satellite: ISO, Copyright: ESA



In the ISM: Kemper et al, 2004.

# Nature and degree of crystallinity of silicates

	Silicate type	Structure
Old stars	Olivine Pyroxene	Amorphous ~ 15 %
Diffuse ISM	85% Olivine + 15% Pyroxene	< 0.4 %
Proto-stars	Pyroxene	Amorphous
proto planetary disks	Pyroxene type	20-25 %

## Subsequent questions....

- Why are there no crystalline silicates in the ISM?
- Since the silicates are amorphous in the ISM, how do we explain the presence of crystalline silicates in the young stars? comets? IDPs?
- How do we explain the chemical modifications of silicates in different environments.
- Is it possible to reproduce some of the microstructure of the IDPs in the laboratory.

# Methodology@IAS

From astronomical observations and analyses of IDPs in the lab



Laboratory experiments

- Synthesis of analogs
- Irradiations (IS), Thermal annealings (CS)
- Characterization



Comparison lab results/observations

**IR**  $\Rightarrow$  Direct comparison with astronomical data and with IDPs (natural dust analyzed by IR microspectroscopy)  
**+ TEM, AFM**  $\Rightarrow$  microstructure, chemical analyses.



# I/ Ionic irradiations

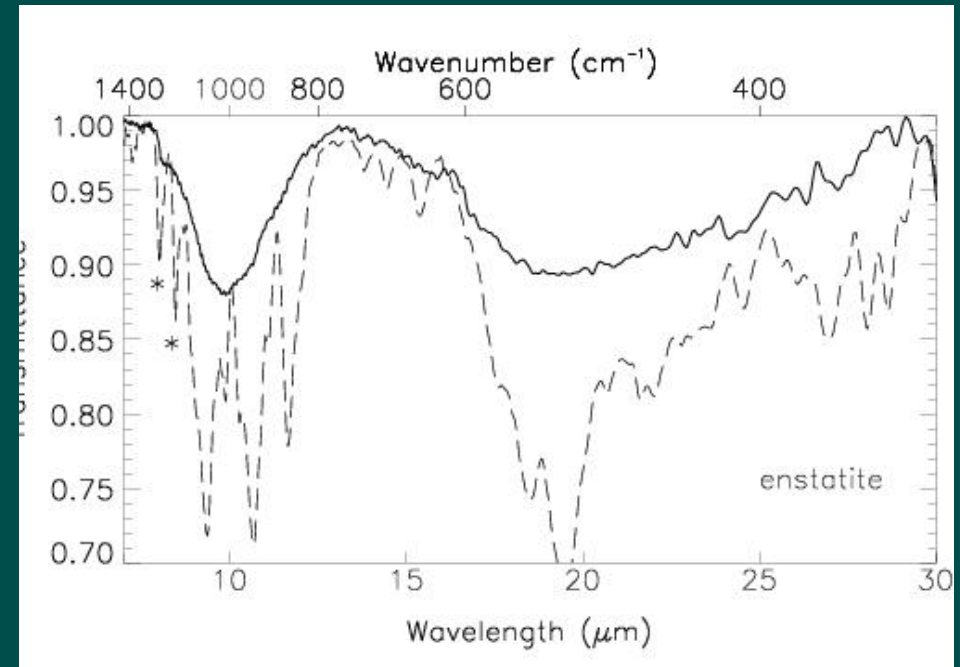
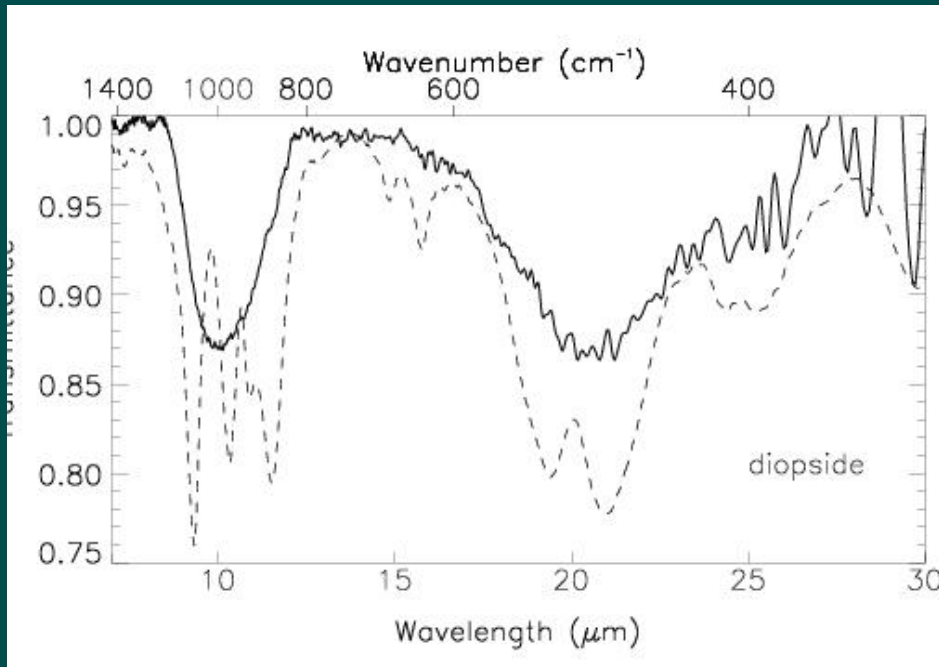
## Experimental conditions

- Silicates: Olivine: Forsterite  $\text{MgSiO}_4$   
Pyroxene: Enstatite ( $\text{Mg,Fe,SiO}_3$ ), Diopside ( $\text{CaMgSi}_2\text{O}_6$ )
- TEM sections , powder embedded in CsI.
- Irradiations:  $\text{He}^+$  et  $\text{H}^+$  at energies few keV and fluences  $\sim 10^{16}$  - $10^{18}$  ions/cm<sup>2</sup>
- IR spectroscopy and TEM analyses

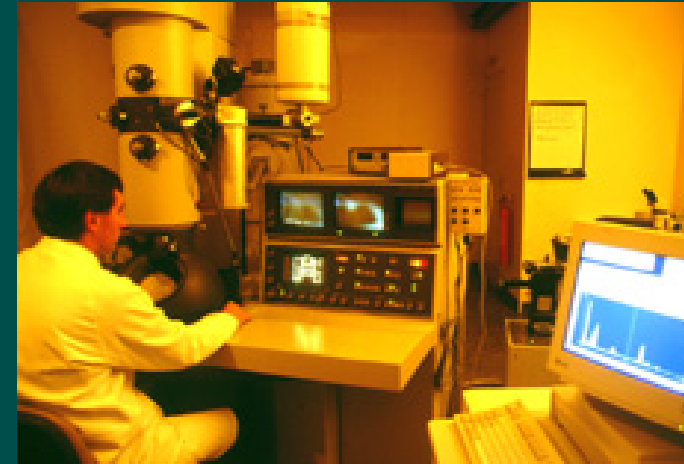
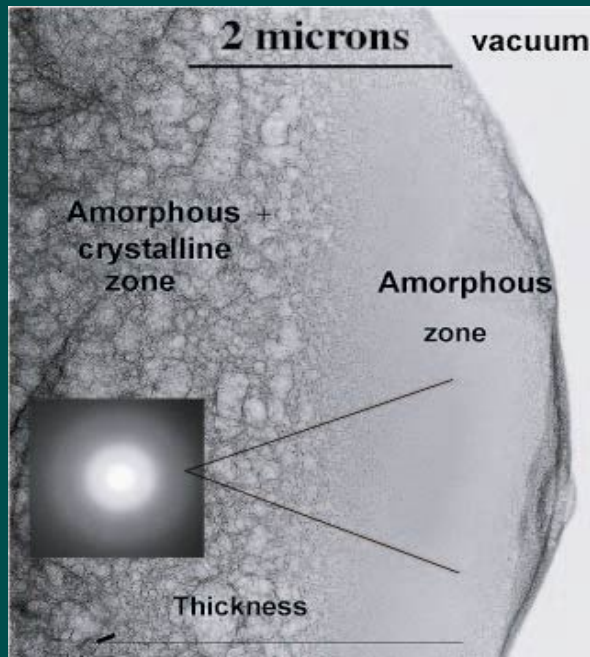
# I-a/ Ionic irradiations: IR spectra

Enstatite, diopside

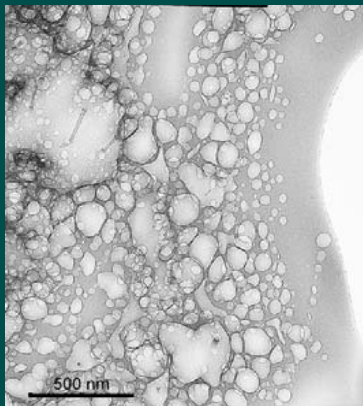
IR spectra obtained before (---) and after (—) irradiations



# I-b/ Ionic irradiations: TEM results



*TEM: Lille*



*TEM view of an olivine sample irradiated with He<sup>+</sup> ions at 10 keV and a fluence of 10<sup>18</sup> ions/cm<sup>2</sup>*

## Microstructural & chemical study

- Amorphisation
- Composition modification  
(loss of Mg & O)
- Induced porosity (bubbles)

K. Demyk (IAS:2000), Ph. Carrez (Lille: 2002)

## Summary of principal results of irradiation:

Olivine: Demyk et al. 2001, Carrez et al. 2002: Irradiations He<sup>+</sup>, 4-10 keV

Pyroxene: Demyk et al. 2004: Irradiations H<sup>+</sup> (10 keV) et He<sup>+</sup> (20-50 keV)

**Irradiation in ISM: Amorphisation + chemical modifications.**

*So.....*

*How do we explain the presence of the crystalline silicates in comets, in DPs, in protoplanetary discs. In quite cold environments in general?*

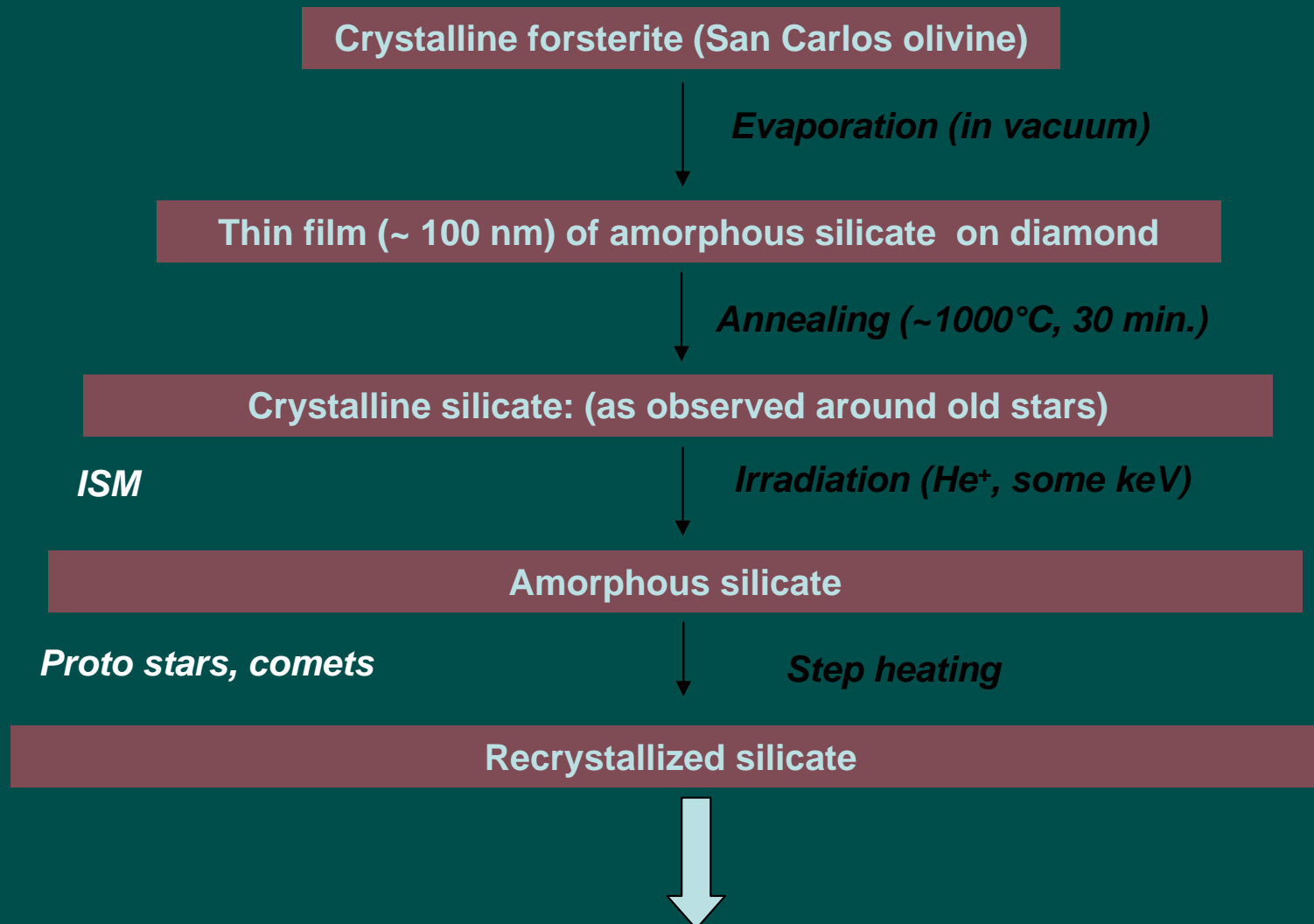
Hypothesis:

Irradiation = ionic implantation ⇒

Modification of the thermodynamical properties of the sample

Experiments for the determination of the re-crystallization activation energy of pre-irradiated samples

# II/ Thermal annealing: Experimental protocol



**Re-crystallization activation energy**

**All the steps are monitored by IR spectroscopy**

# Analog synthesis



$\text{Mg}_{1.8}\text{Fe}_{0.2}\text{SiO}_4$

## Advantages:

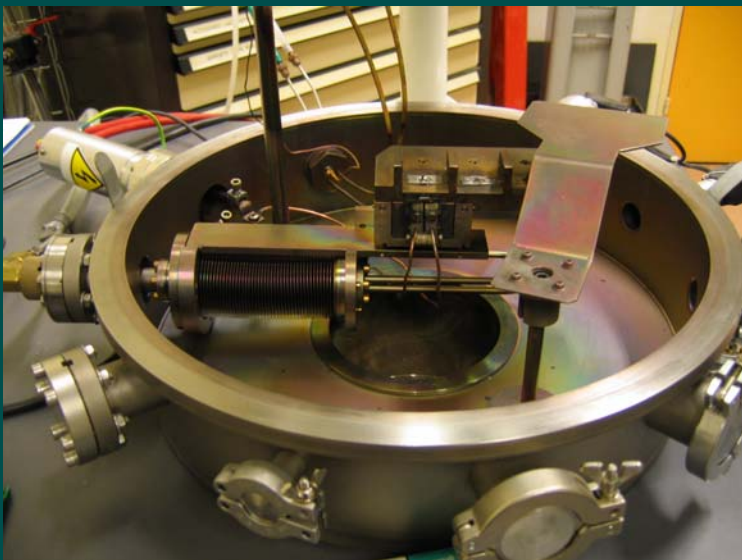
- Thin film ~ 100 nm on a diamond disc ~ 3 mm diameter  $\Rightarrow$  High surface-to-volume ratio.
- **Diamond:** resists to high temperatures and transparent to IR in the silicate regions.

## Disadvantage

- impossible to couple IR measurements with TEM analyses.

## Solution:

- remove the film from the diamond to a suitable substrat for TEM.



# Annealing set-up

In a tubular furnace under vacuum ( $\sim 10^{-7}$  mbar)



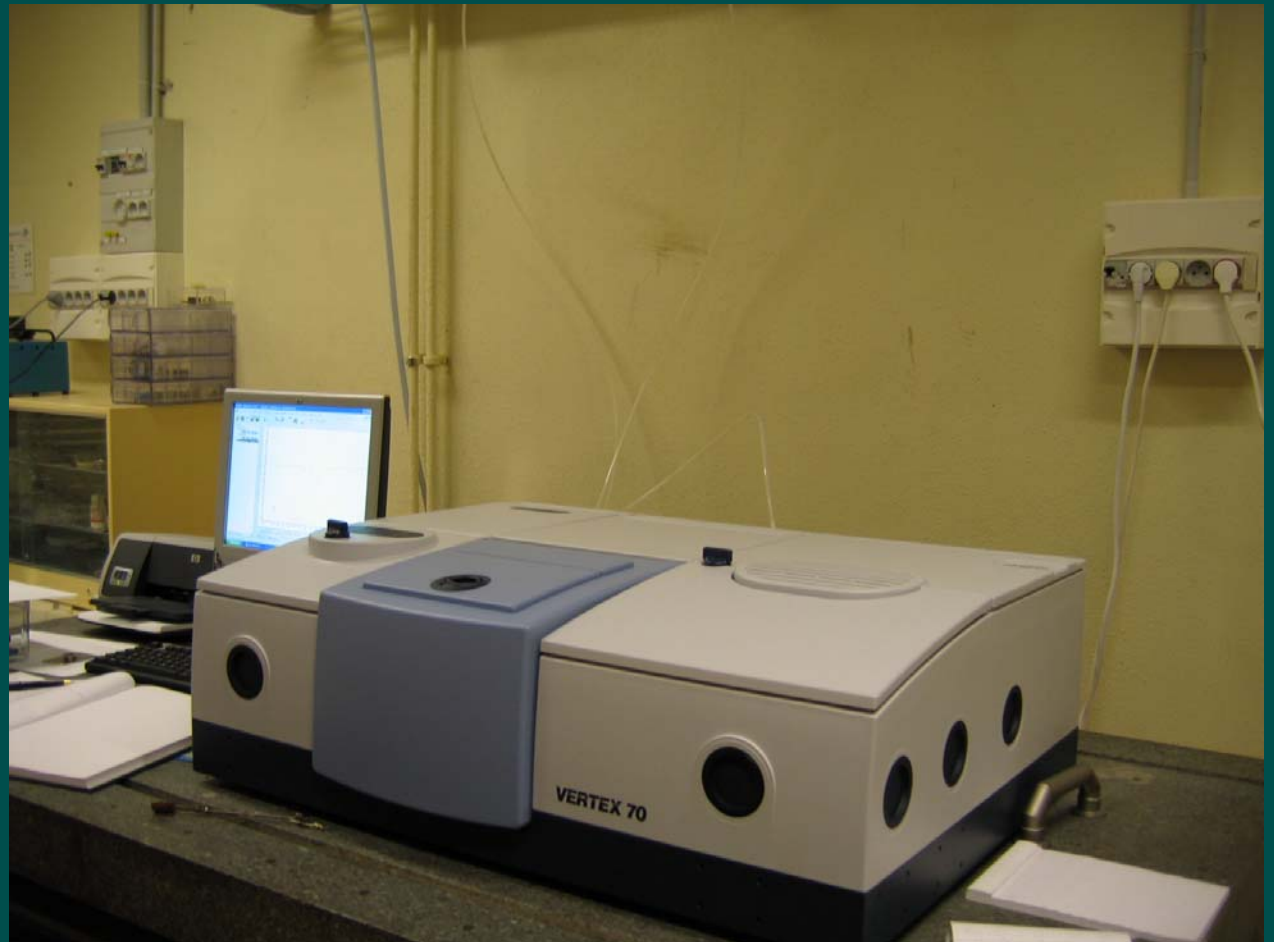
Aim: Simulation of the interstellar amorphous silicates incorporation in the protoplanetary disks.

# IR spectroscopy

IR spectroscopy by transmission

[7000, 400]  $\text{cm}^{-1}$

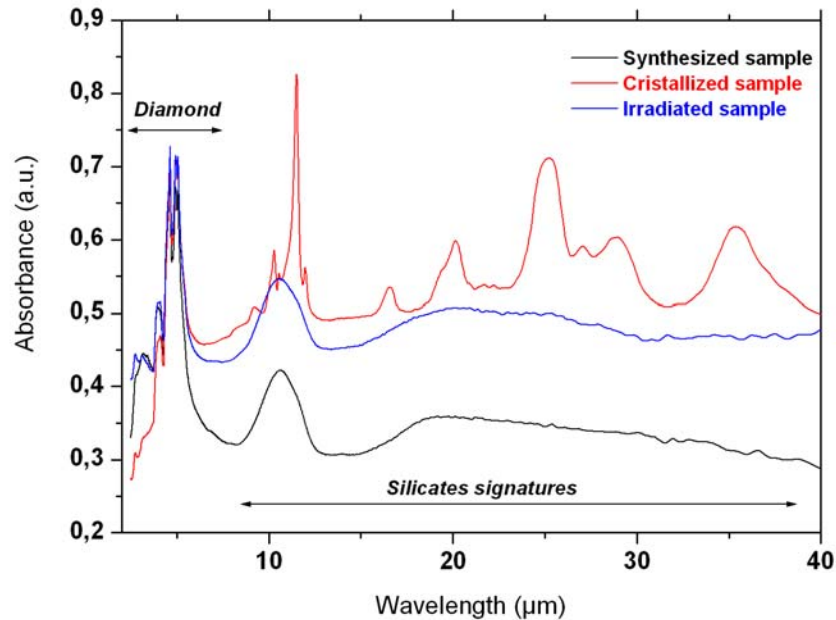
1.5-25  $\mu\text{m}$



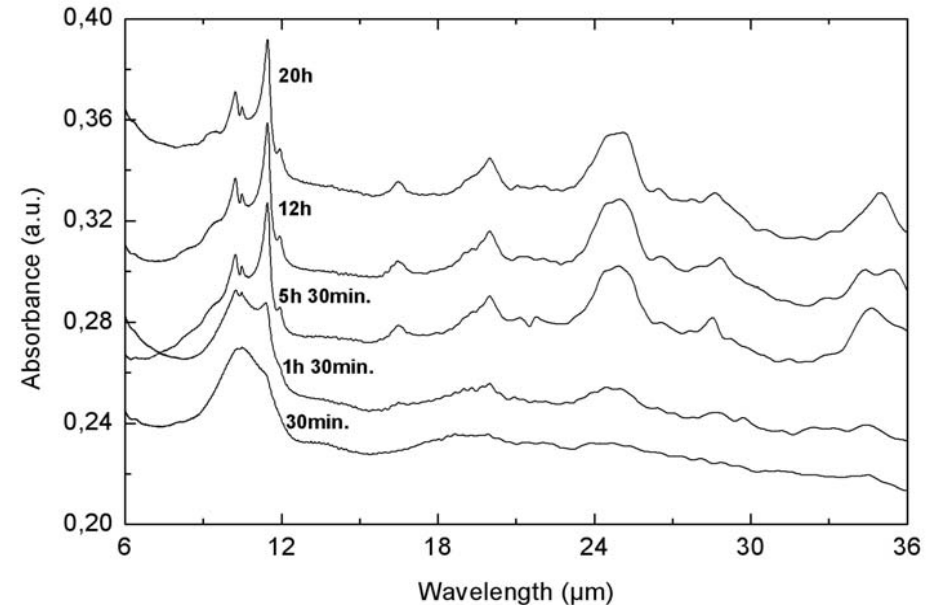


# II-a/ Thermal annealing: IR spectra

Typical IR spectra



Bands evolution with annealing time: 750°C



$$t = \nu^{-1} \exp\left(\frac{E_a}{kT}\right)$$

Fabian et al. (*A&A*, 364, 282, 2002)

39100 K

Brucato et al. (*Planet. Space Sci.*, 50, 829, 2002)

40400 K

Djouadi et al. (*A&A*, 440, 179, 2005)

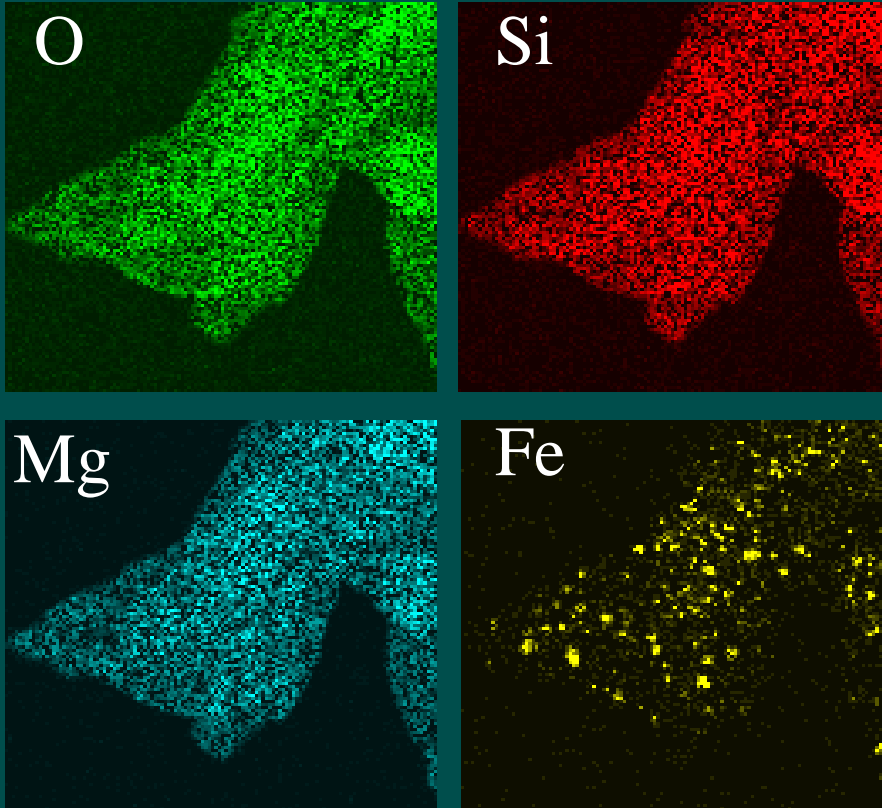
(41700 ± 2400) K

⇒ *The crystallization of silicate dust is independent of its history.*

⇒ *The crystalline silicates observed in the vicinity of cold environments (around young stars and some comets) have probably been crystallized before their injection into these environments.*

## II-b/ Thermal annealing: TEM analyses

300 nm

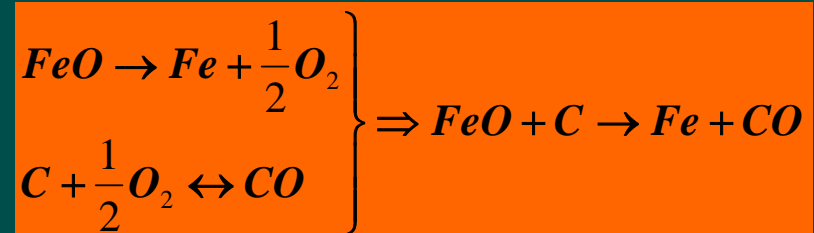


1/ Presence of metallic beads of ~ 2-50 nm.

2/ Formation of amorphous silicates free of iron. (in accordance with the observations)

3/ For the samples partially crystallized: formation of forsterite crystals+Fe beads.

⇒ The Fe initially present in form of FeO has been reduced.



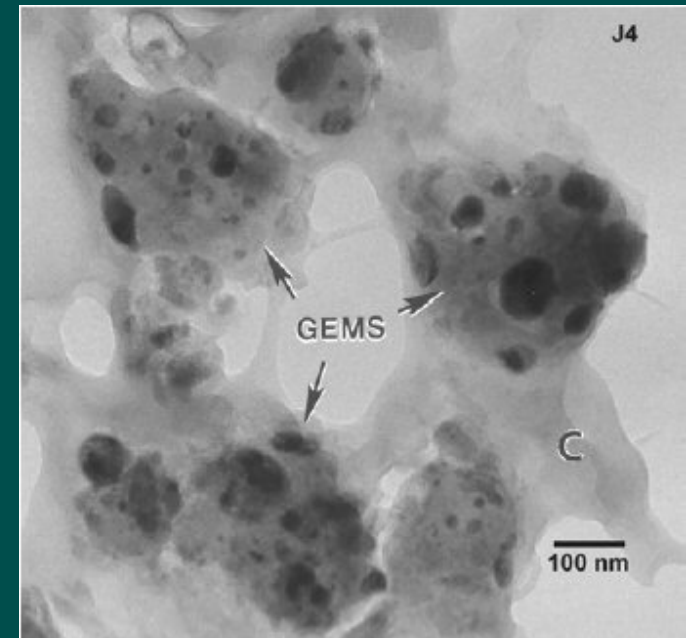
## Summary of principal results of annealing:

- Crystallization enthalpy is not modified after ionic irradiation ⇒ Redistribution of the matter between inner hot regions and outer cooler ones.
- Formation of forsterite (Mg rich silicates) in accordance with the observations
- Locking iron as metallic particles within the silicates explains why astronomical silicates always appear observationally Fe-poor (metallic Fe has no IR signature)

# Astrophysical implications:

## a/ GEMS formation scenario

- GEMS= Glass Embedded with Metal and sulfides: 'intriguing' metallic inclusions in anhydrous IDPs.
- No consensus to explain their origin, and their formation process.
- We propose a possible simple scheme for their formation : they are probably modified in the protoplanetary disks in reducing conditions.



# Astrophysical implications:

## b/ Grain alignment (?)

- Polarized signal of the starlight  $\Rightarrow$  IS grains are aspherical and aligned by a local magnetic field
- 1951: DG model of spinning interstellar grains but considering the random collisions with molecules, gas atoms.. Impossible to maintain the grain alignment in a  $\sim \mu\text{G}$ .
- 1969: Jones & Spitzer and 1978: Duley argued DG efficient if « Fe inclusions» are present. SPFM alignment.
- 1985: Mathis. IS grains = aggregates of tinier particles, some of them SPFM.
- 1995: P.G. Martin, GEMS = solution to this old and mystery problem..
  - Fe(Ni)S inclusions (due to irradiation (Bradley 1994)) = SPFM.
  - Any C-rich IS grain wouldn't be aligned (Ok with Mathis 1986).
  - GEMS are aspherical 1.4:1 (kim & Martin 1995)

**The problem of IS grains alignment is still unresolved !!.**

**We propose that these grains contain appreciable amounts of metallic Fe nano-phase**

# Summary

- Experimental set-up to process synthetic grain materials
  - Simulation in the laboratory of interstellar and circumstellar physical conditions
    - ⇒ Reproduction of some physical and structural properties of dust
- Formation of Fe-Ni beads (GEMS-like structure)
  - ⇒ Possible explanation for the grain alignment.

**The magnetic susceptibility measurements in our sample are under progress**

The end, and .....

Thanks for your attention !!