Non-Evaporable Getter Thin Film Coatings for Vacuum Application

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Supervisor: Paolo Chiggiato

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Non-Evaporable Getter Materials Introduction

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Non-Evaporable Getter Materials

Non-Evaporable Getter Materials

CAPTURE PUMPS →

Non-Evaporable Getter Materials

CAPTURE PUMPS → they remove molecules from the gas phase by fixing them on their surface

$$\tau = \tau_0 \exp\left\{\frac{E}{k_B T}\right\}$$

Sojourn Time

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Sojourn Time

NEGs chemically react with the gases present in vacuum system

- free of contaminants
- free of the oxide layer

Non-Evaporable Getter Materials Introduction

Active Surface →

Active Surface → heating in situ the NEG thin film coating in order to allow the diffusion of the oxygen into the bulk material in vacuum system

Active Surface

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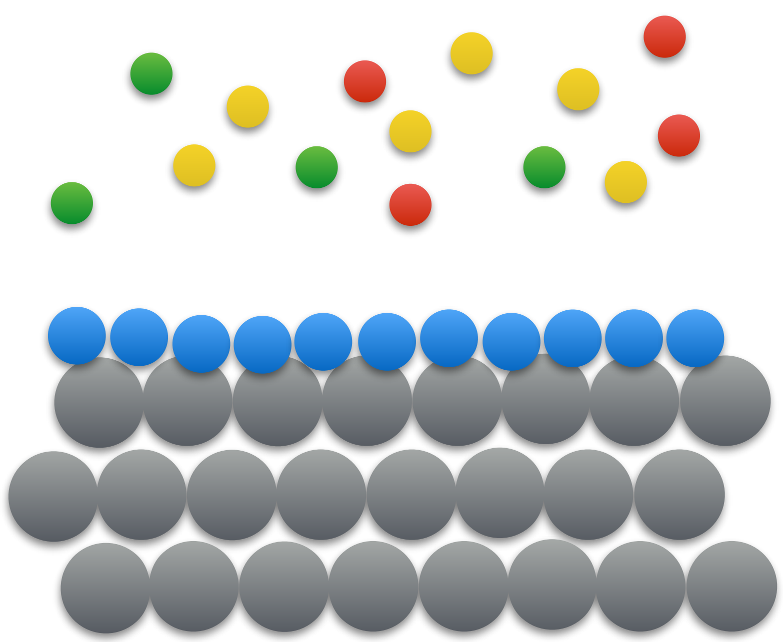

Room Temperature

Non-Evaporable Getter Materials Introduction

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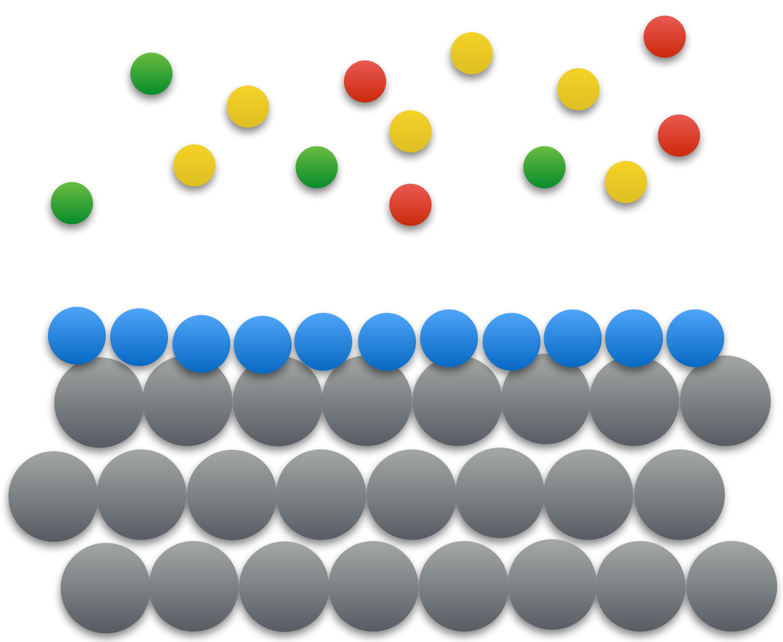

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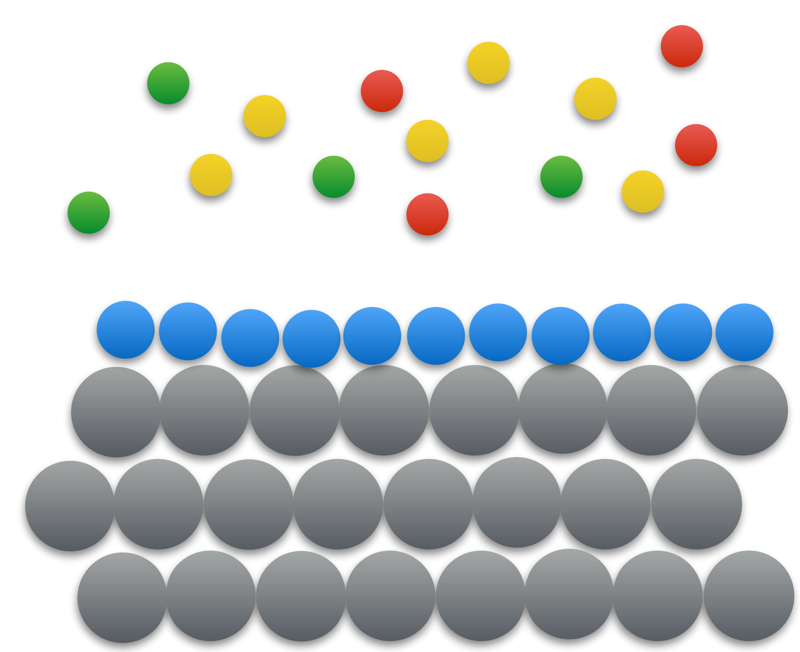

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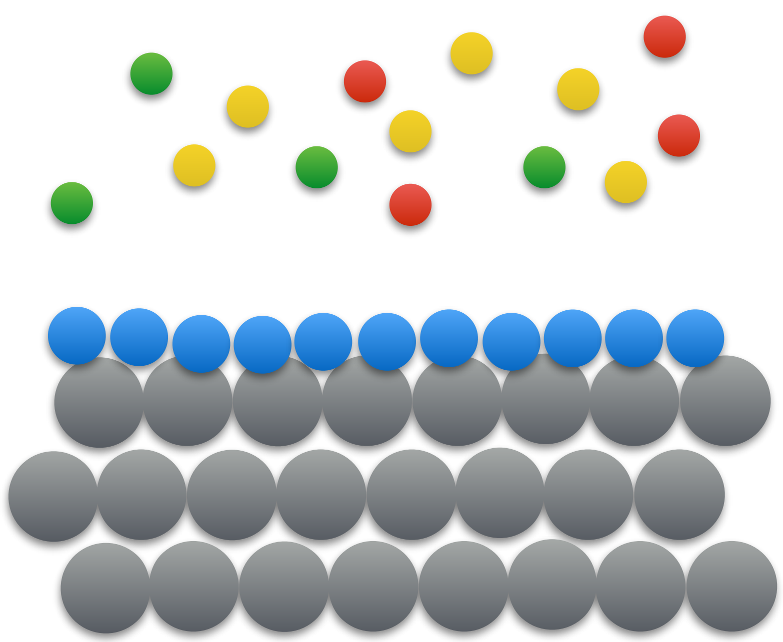

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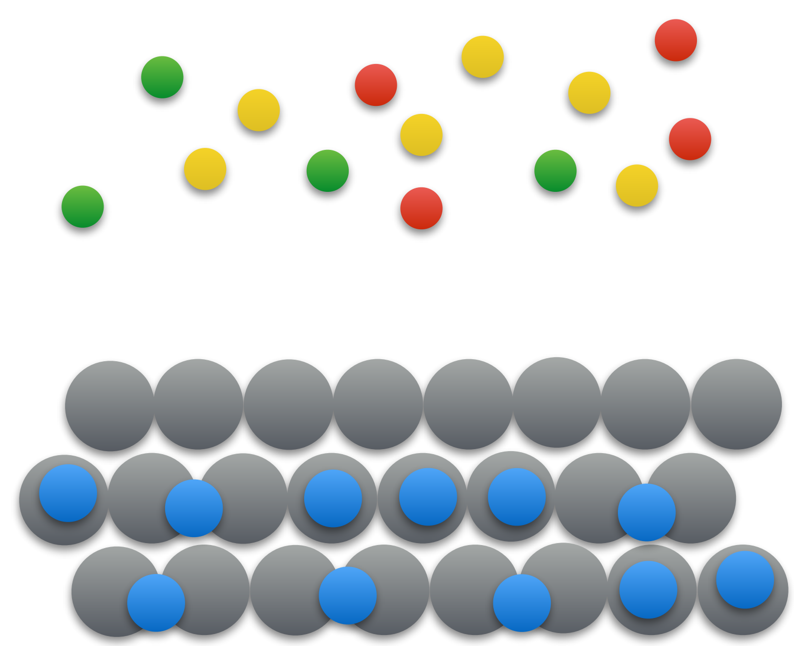

Activation Temperature

Non-Evaporable Getter Materials Introduction

Active Surface

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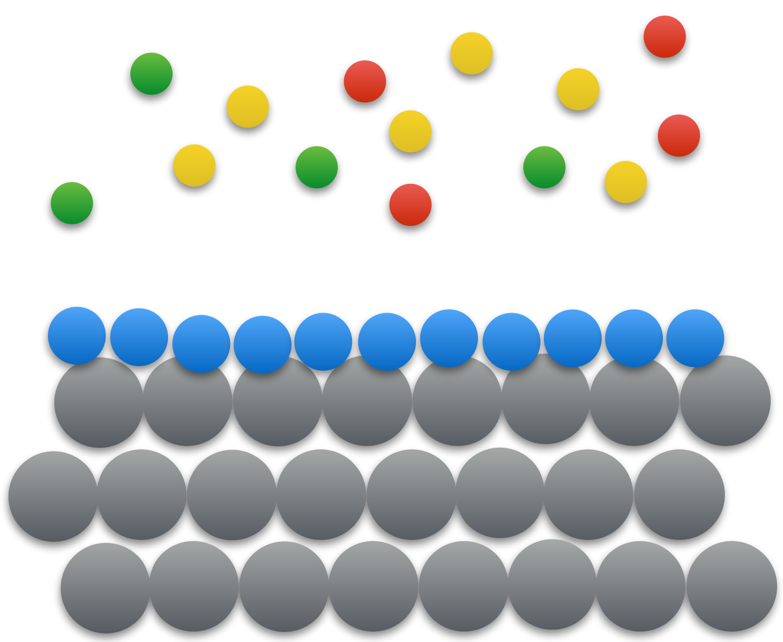
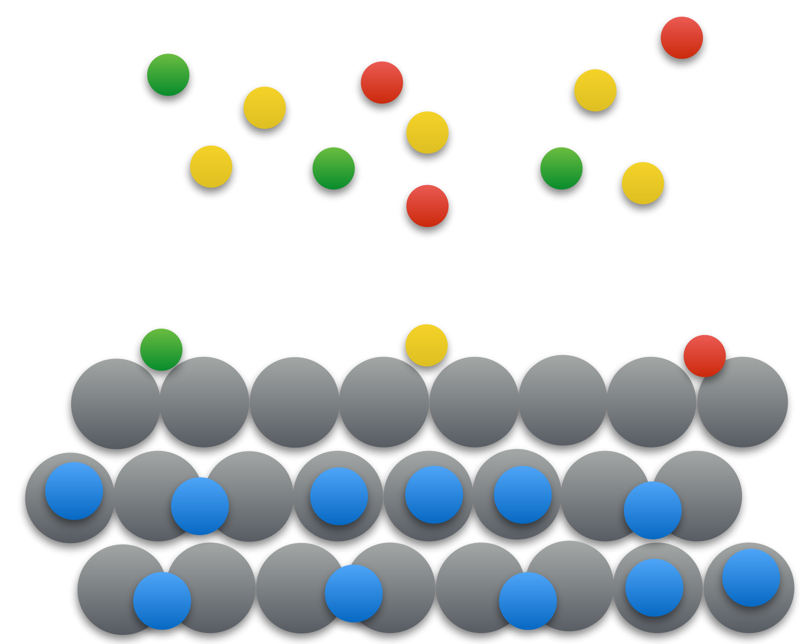


Room Temperature



Active Surface → heating in situ the NEG thin film coating in order to allow the diffusion of the oxygen into the bulk material in vacuum system

- thermodynamically allowed
- reaction fast kinetics

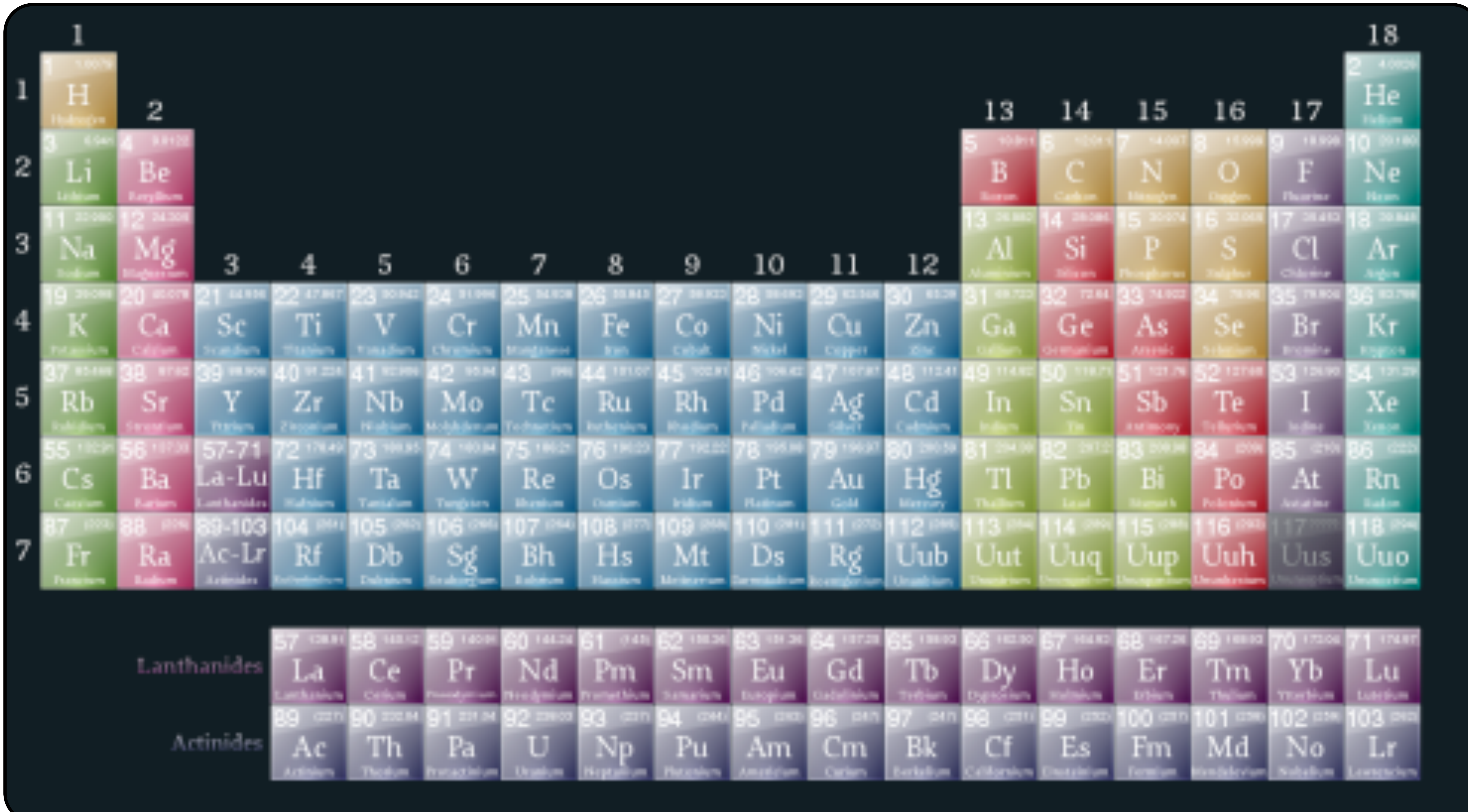
→ **Activation Temperature**

Temperature at which the NEG pumping properties reach their maximum value.

Fundamental characteristics of NEG Material

- Solubility limit for oxygen
- Diffusivity for oxygen
- Reactivity
  - large enthalpy of adsorption for the gases in vacuum chambers
  - hydride phase with low dissociation pressure

Suitable materials



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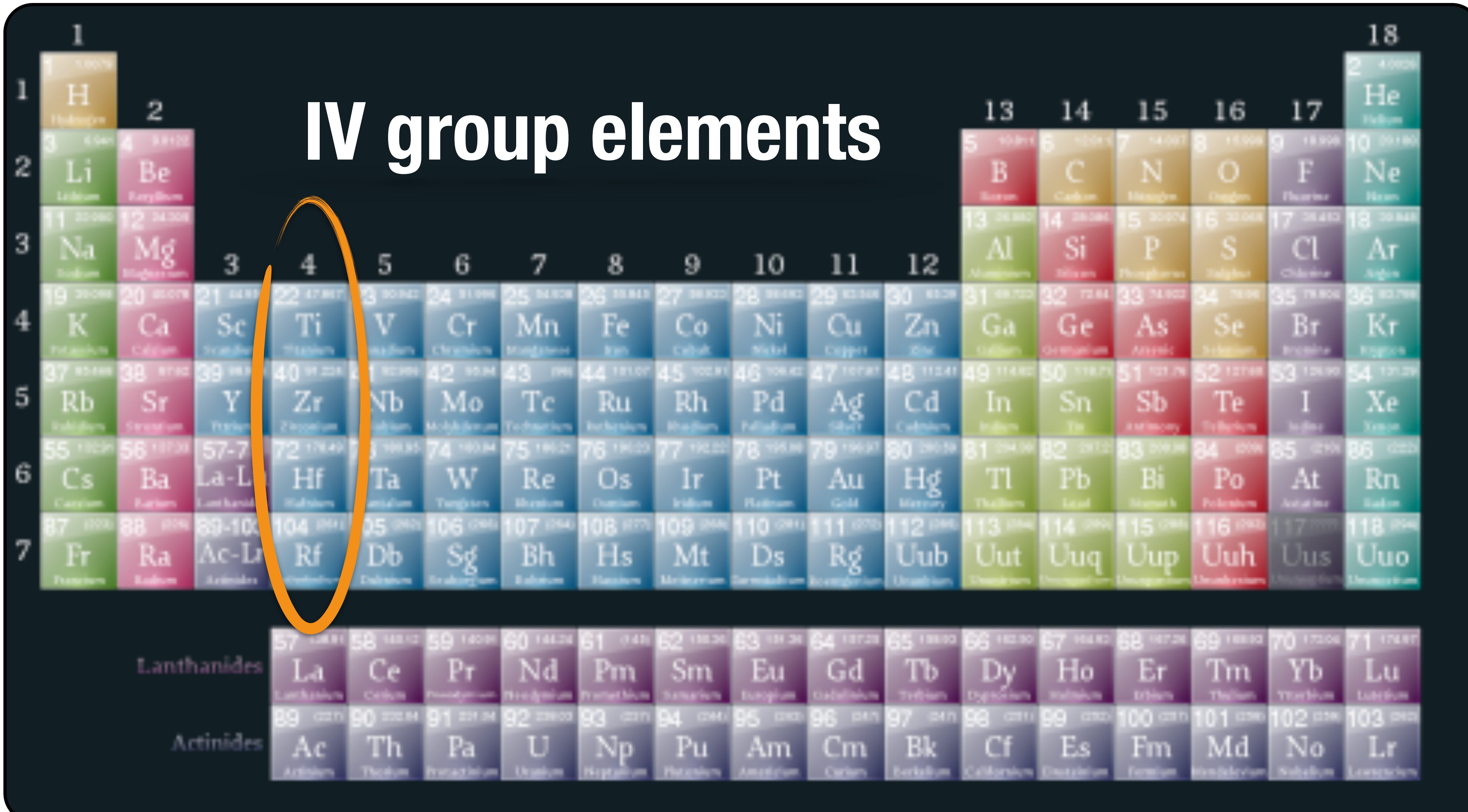

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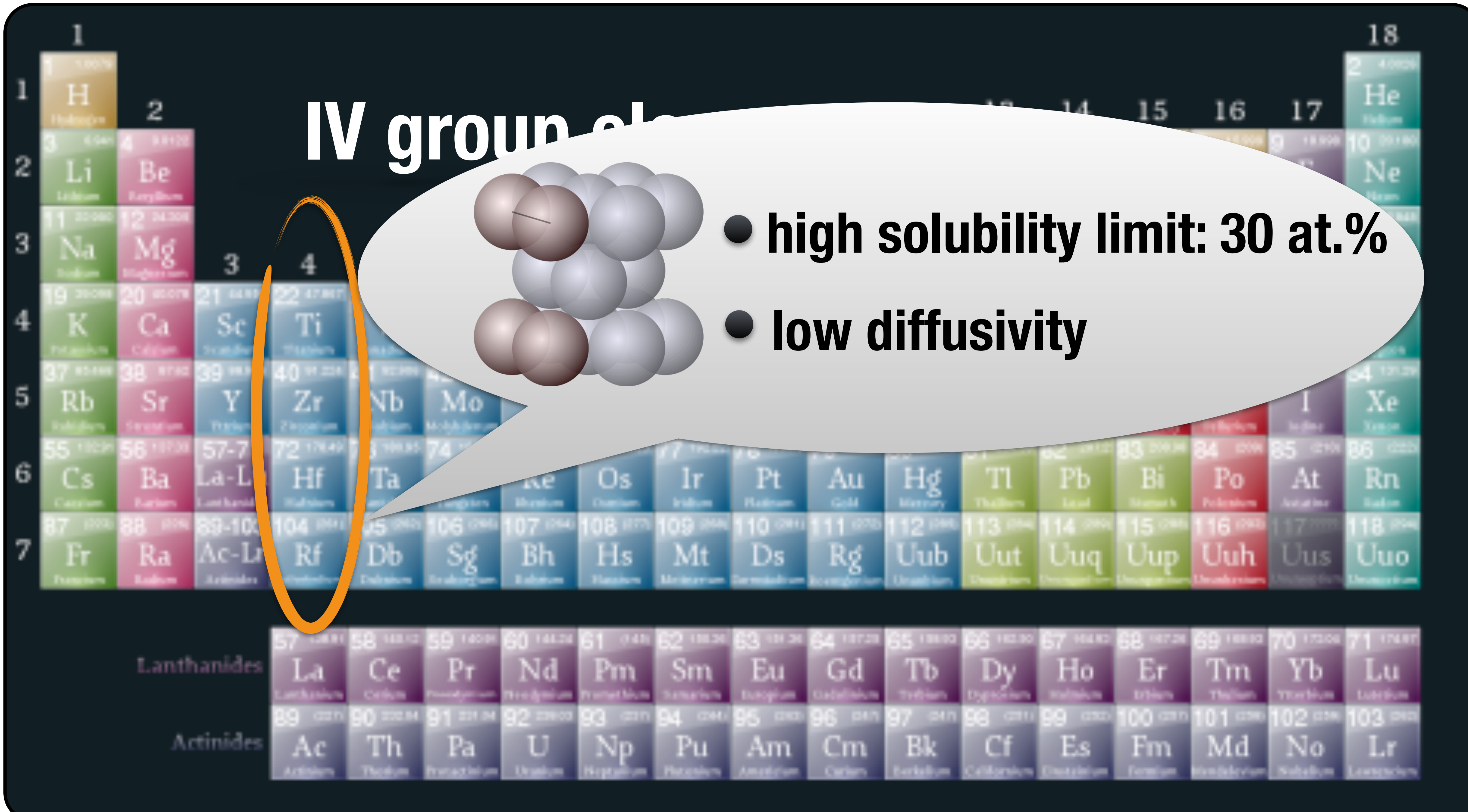

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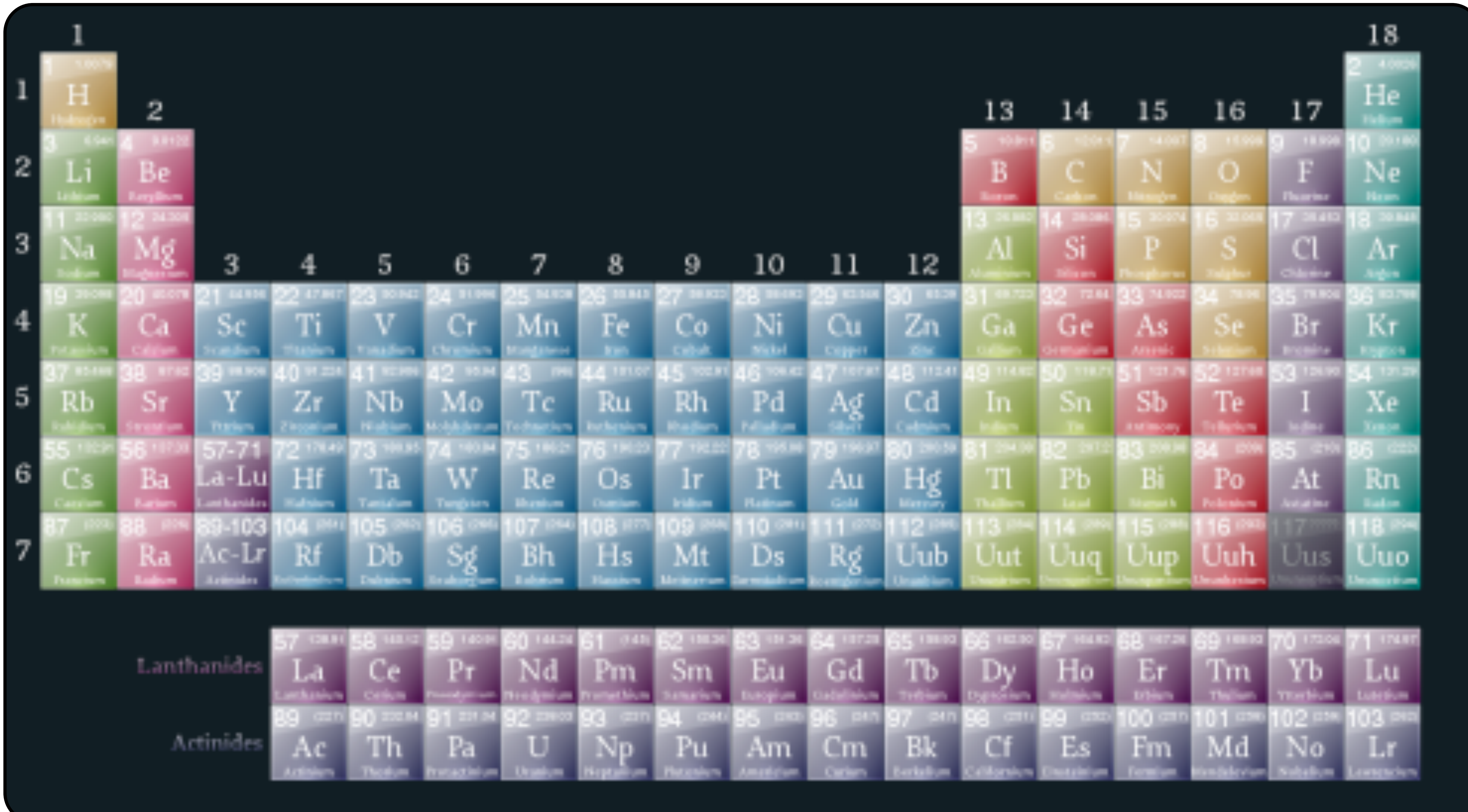

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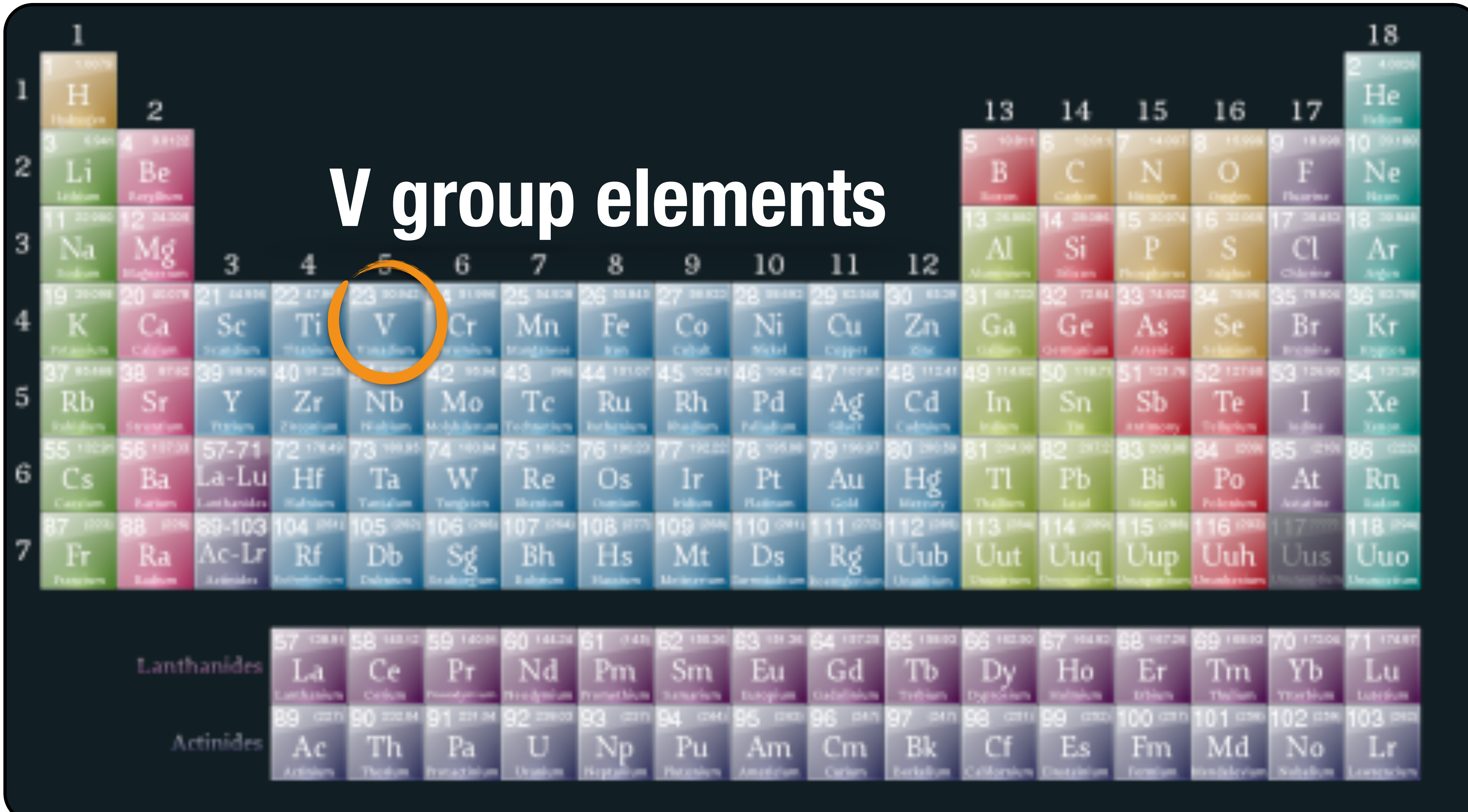

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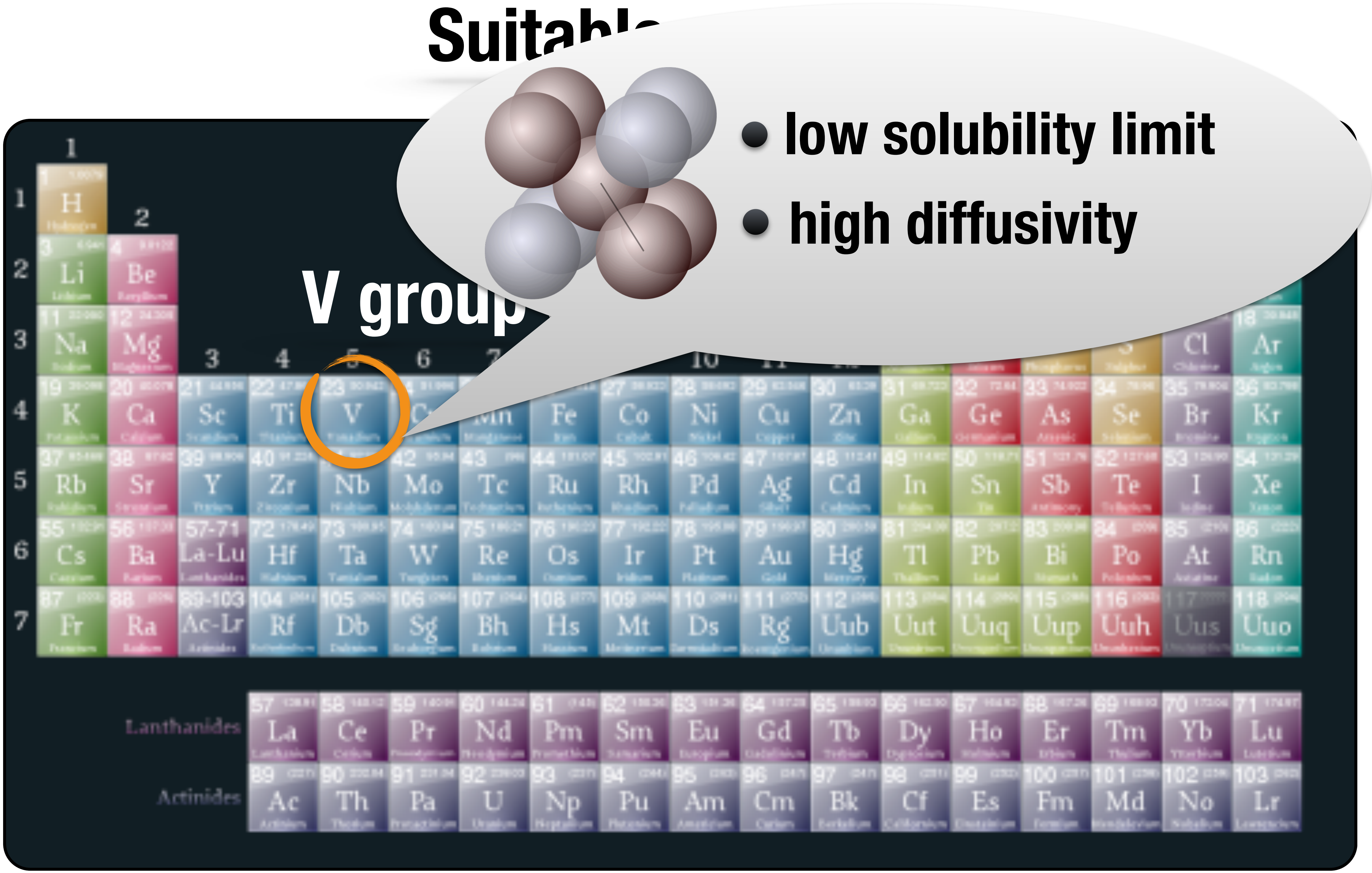
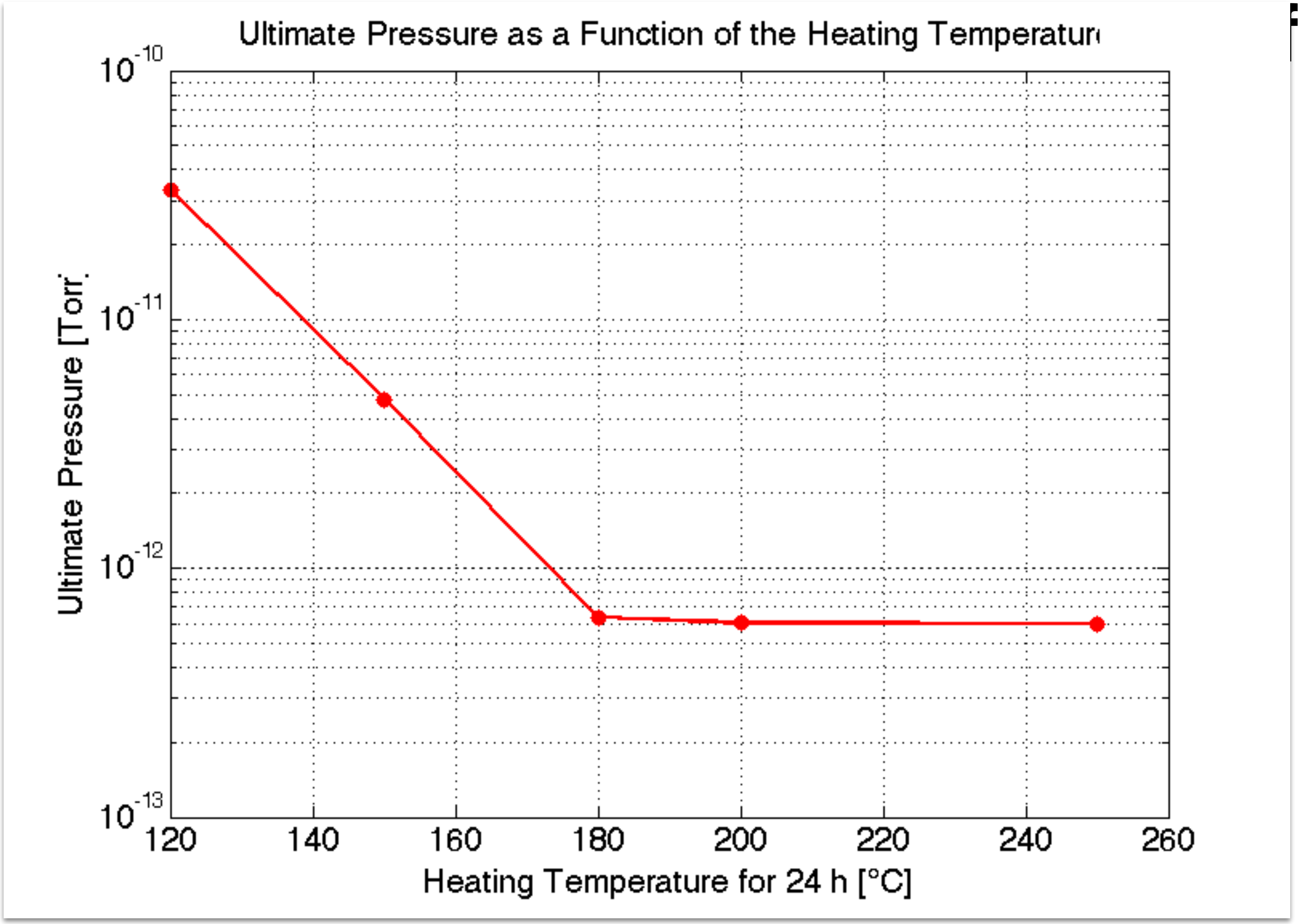


$Ti_{30}Zr_{30}V_{40}$ is fully activated after 24 h of heating at 180 °C

Non-Evaporable Getter Materials Introduction



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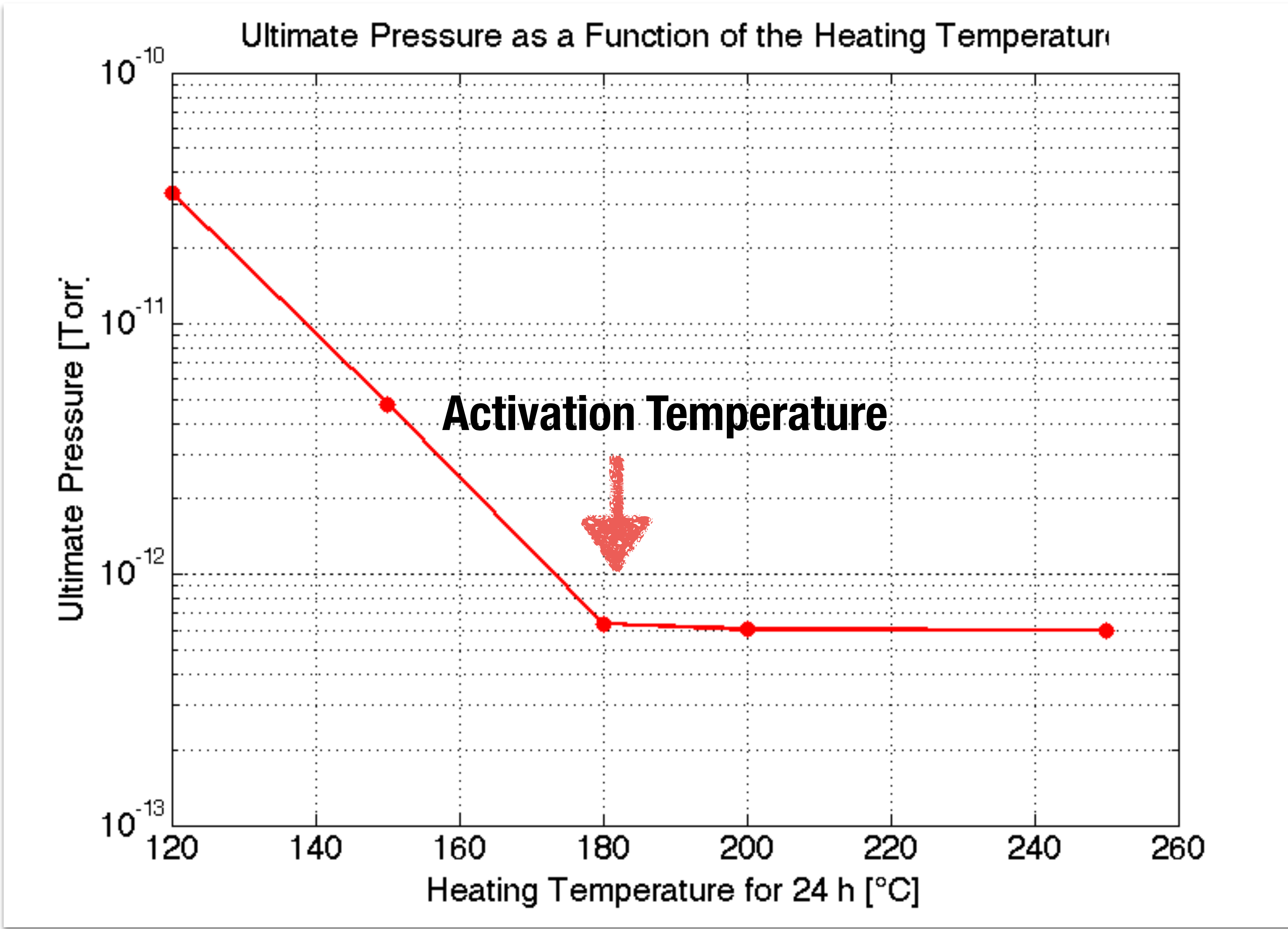

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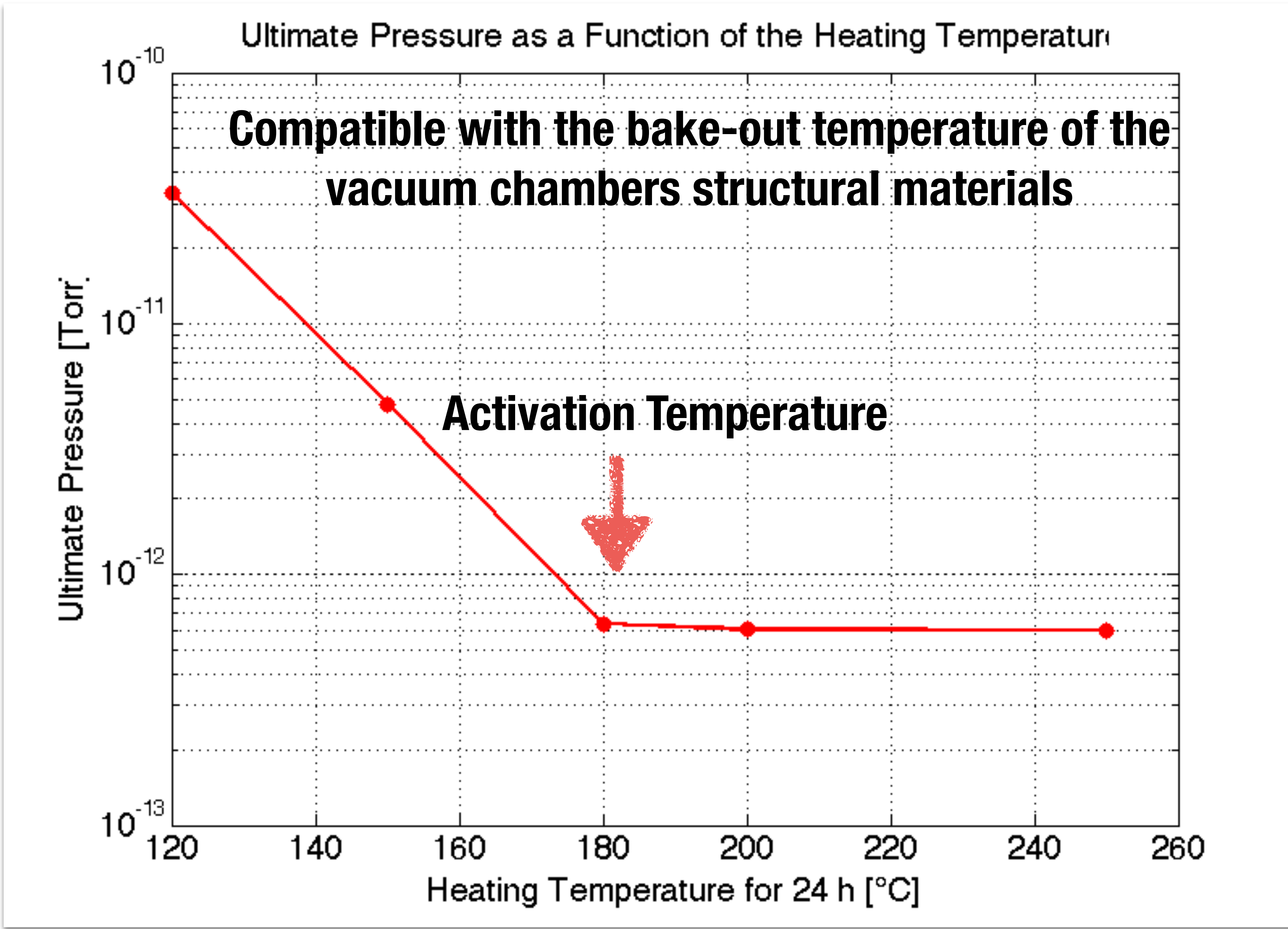


My Work

- What is going to happen to the NEG coated beam-pipes exposed to LHC atmosphere?

My Work

- **What is going to happen to the NEG coated beam-pipes exposed to LHC atmosphere?**

Ageing Campaign

My Work

- What is going to happen to the NEG coated beam-pipes exposed to LHC atmosphere?

  Ageing Campaign

- What is going to happen to the IV group thin film if V is added?

My Work

- What is going to happen to the NEG coated beam-pipes exposed to LHC atmosphere?

  Ageing Campaign

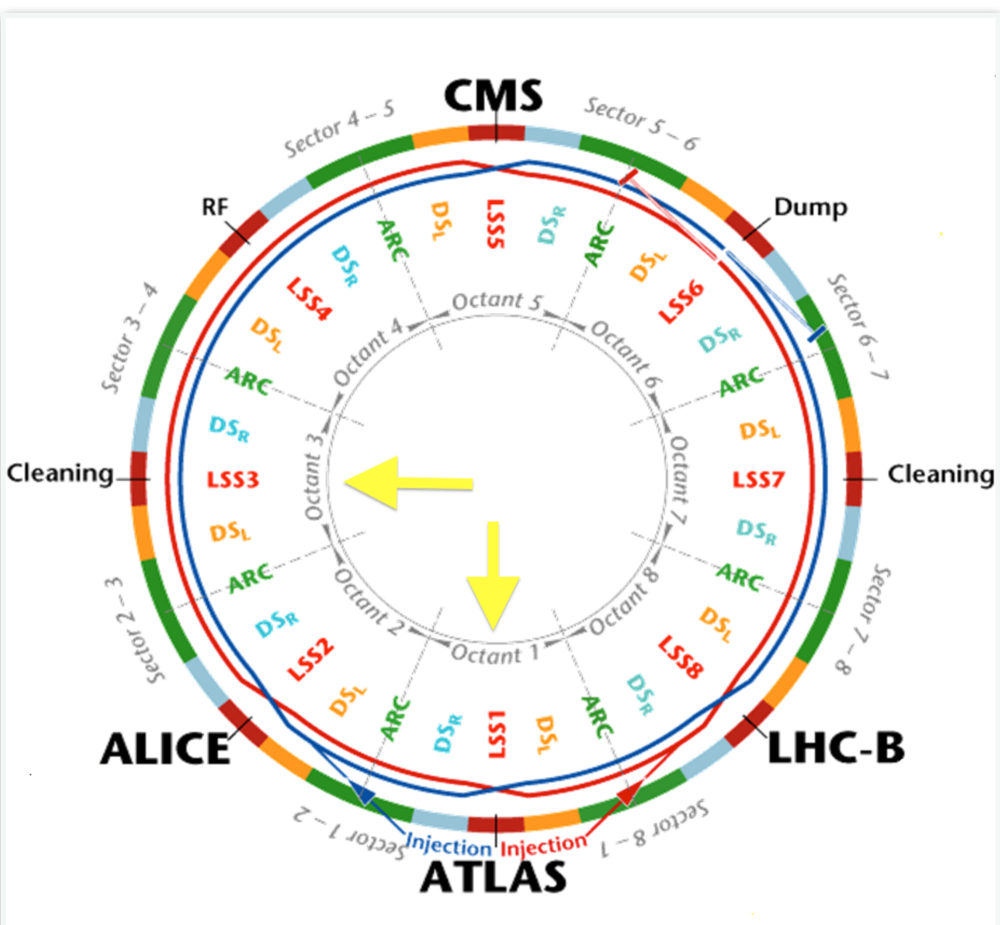
- What is going to happen to the IV group thin film if V is added?

  Coating Campaign

Ageing Campaign

Exposure Points

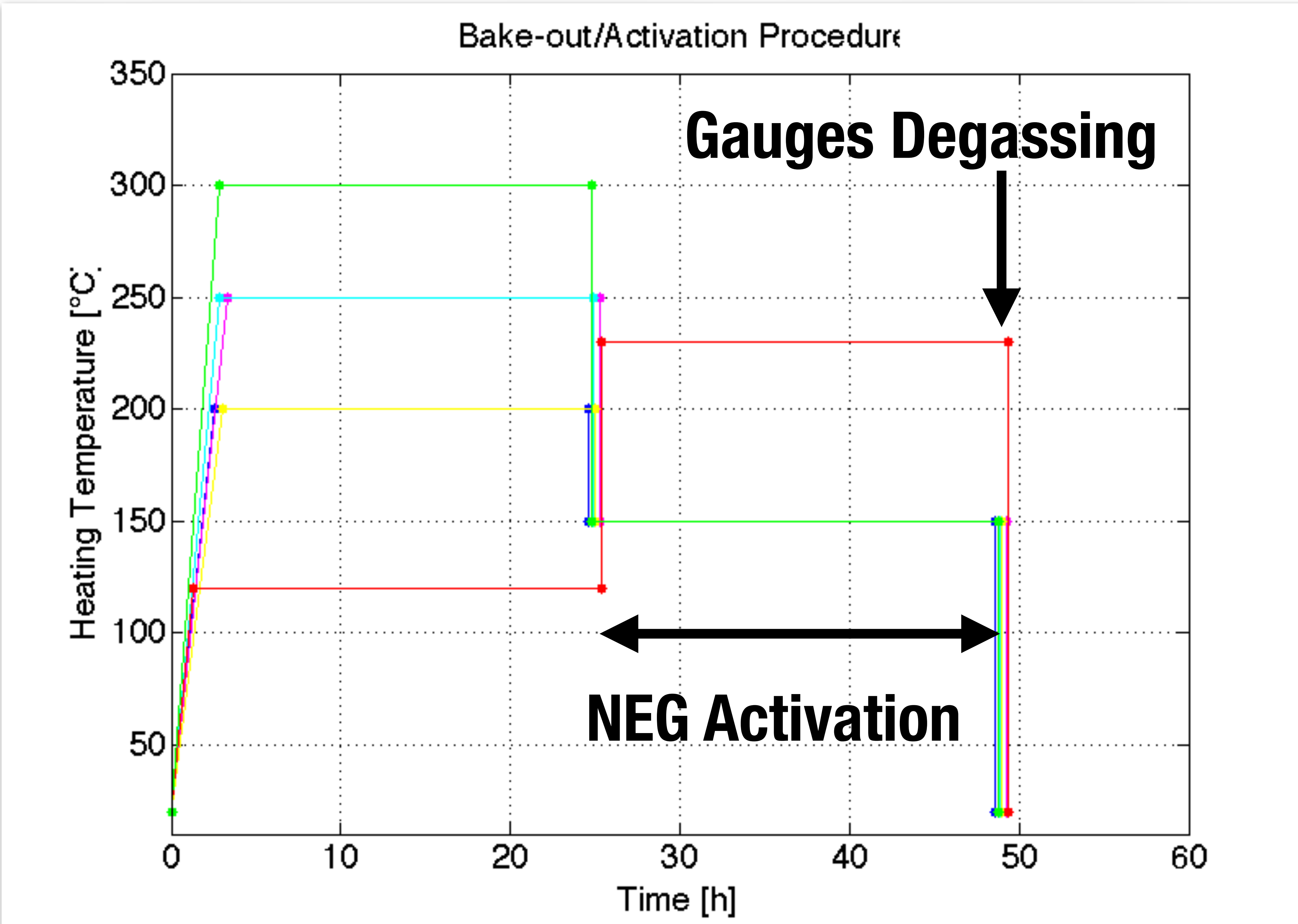
SAMPLE	Exposure Point	Exposure Duration
Reference	none	0 month
LSS3_1	Point 3	1 month
LSS3_2	Point 3	3 months
LSS1_1	Point 1	9 months
LSS1_2	Point 1	
N2 sealed	none	none



Bake-out/Activation Procedure

	STEP 1	STEP 2	STEP 3
Ramp Rate [°C/h]	75	75	80
Temperature [°C]	120	230	20
Duration [h]	24	24	24

Bake-out/Activation Procedure



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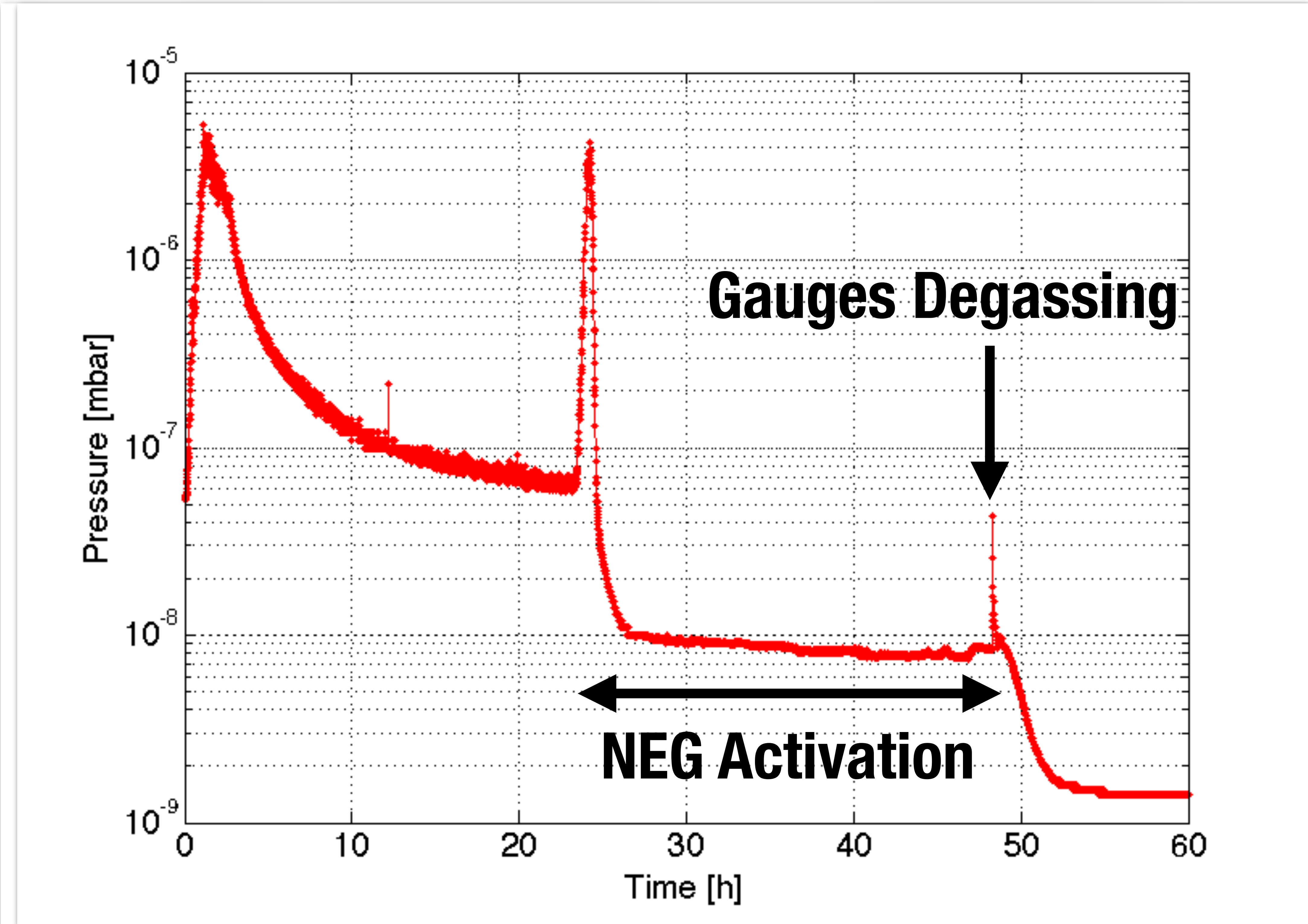
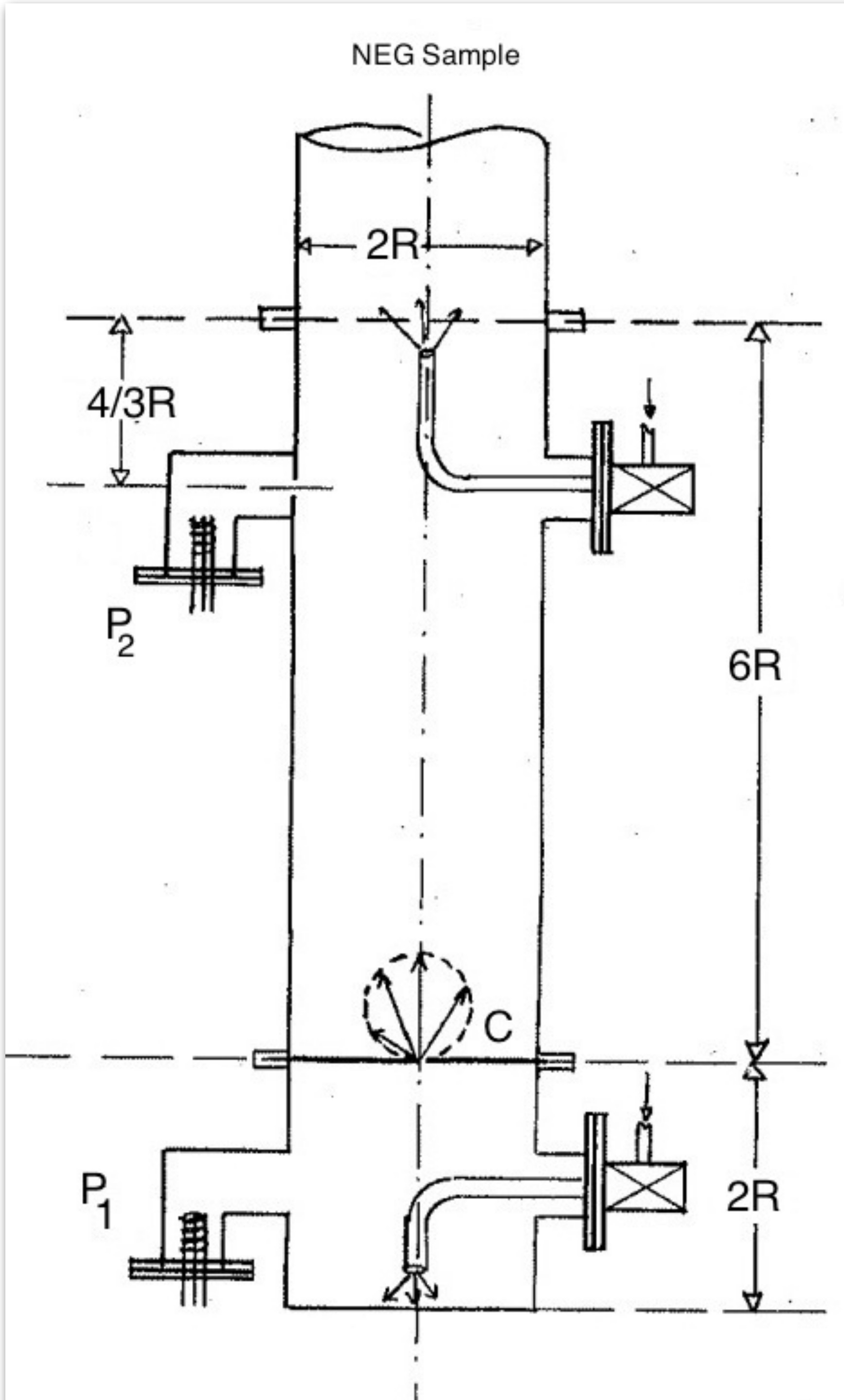


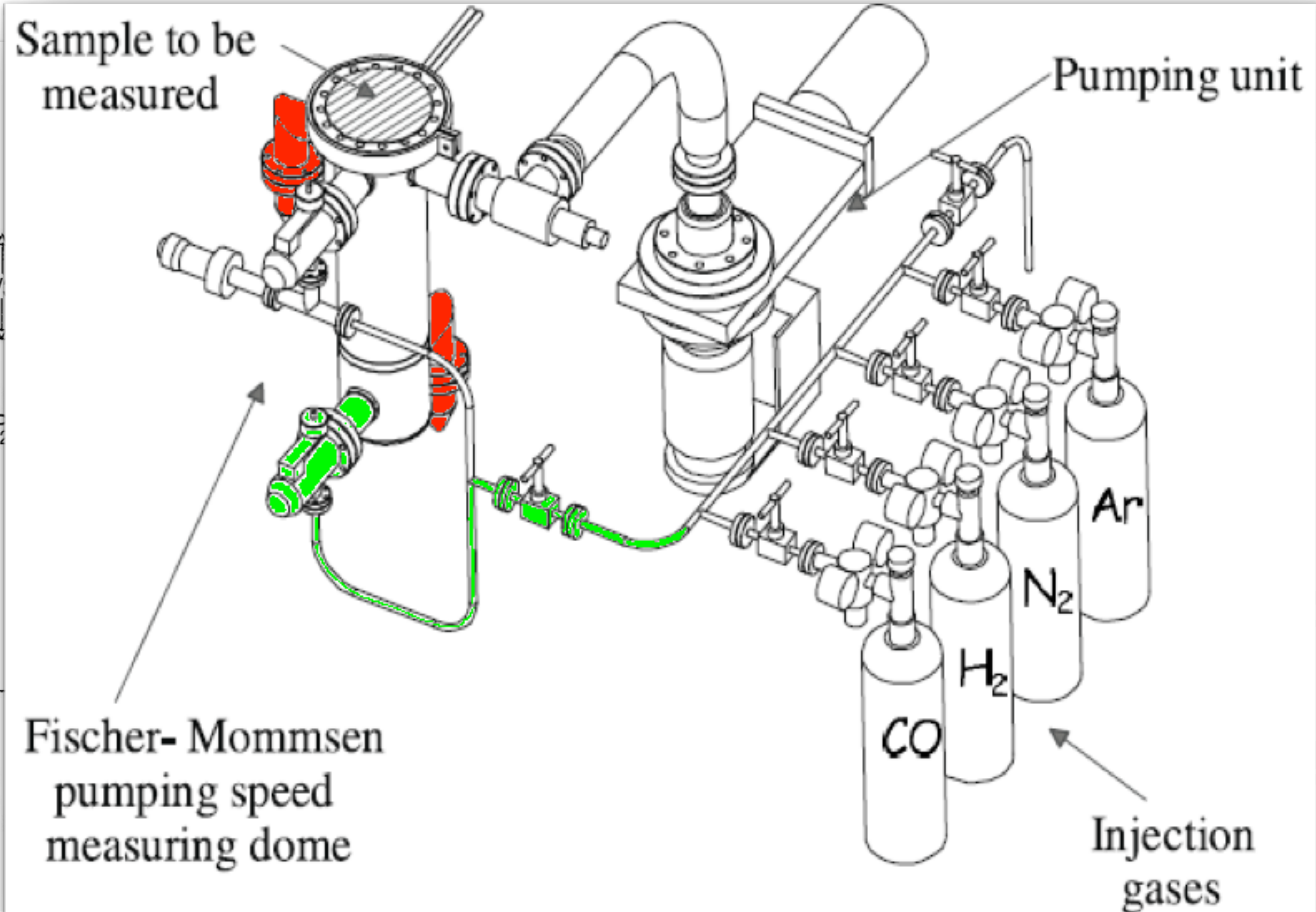
Fischer-Mommsen Dome



$$S_{eff}^{NEG} = C\left(\frac{\Delta P_1}{\Delta P_2} - 1\right) - S_{eff}^{TMP}$$

System Parameters		
	H	CO
S	8	4,37
S	3456	924

Fischer-Mommsen Dome



Experimental Results

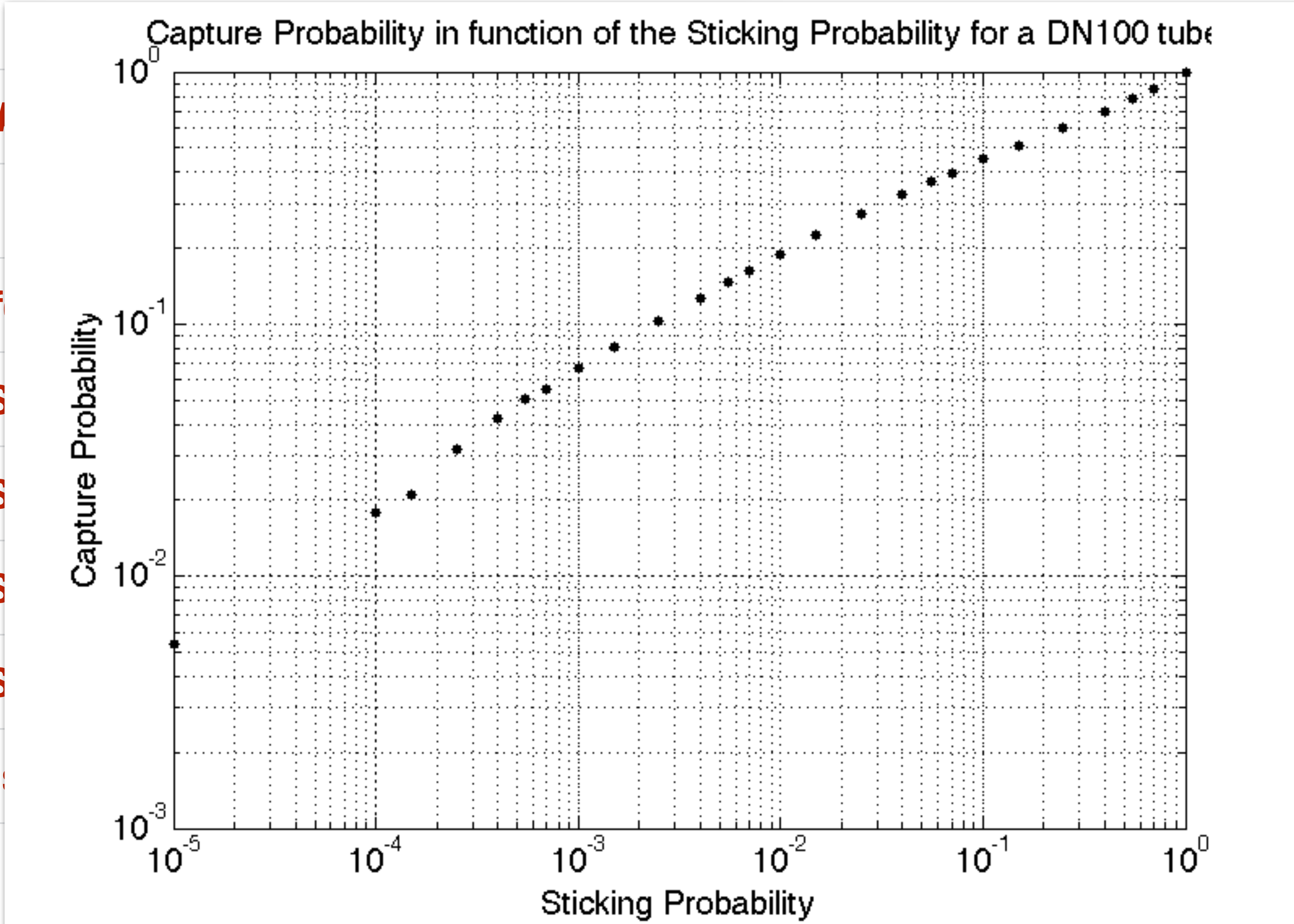
Ageing Campaign

SAMPLE	H2 Pumping Speed [l/s]			Sticking Probability		
	I Act	II Act	Vented	I Act	II Act	Vented
Reference	381	-	-	4 10	-	-
LSS3_1	-	761	386	-	1,5 10	4 10
LSS3_2	395	-	-	4 10	-	-
LSS1_1	615	-	669	1 10	-	1 10
LSS1_2	NOT TESTED					
N2 sealed	NOT LEAK TIGHT					

Experimental Results

Ageing Campaign

SA
Ref
LSS
LSS
LSS
LSS
N2



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SAMPLE	I Activation		II Activation		Vented	
	V1 [Torr]	V2 [Torr]	V1 [Torr]	V2 [Torr]	V1 [Torr]	V2 [Torr]
Reference	7,7 10-11	1,52 10-11	-	-	-	-
LSS3_1	-	-	5 10-11	1,3 10-10	2,65 10-10	9,3 10 -11
LSS3_2	5 10-11	1,1 10-10	-	-	-	-
LSS1_1	1,80 10-9	3,5 10-9	-	-	1,61 10-9	3,6 10-9
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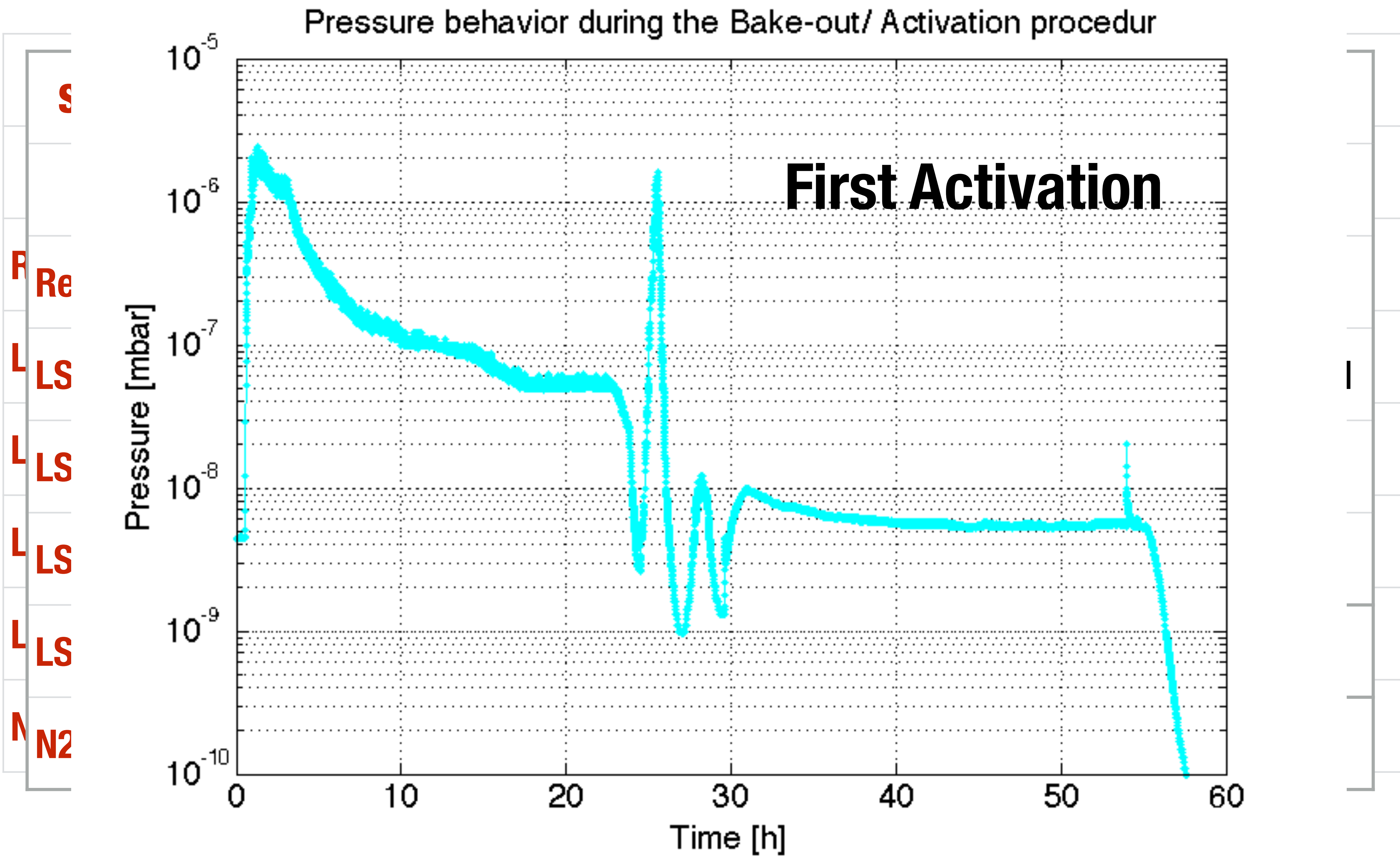

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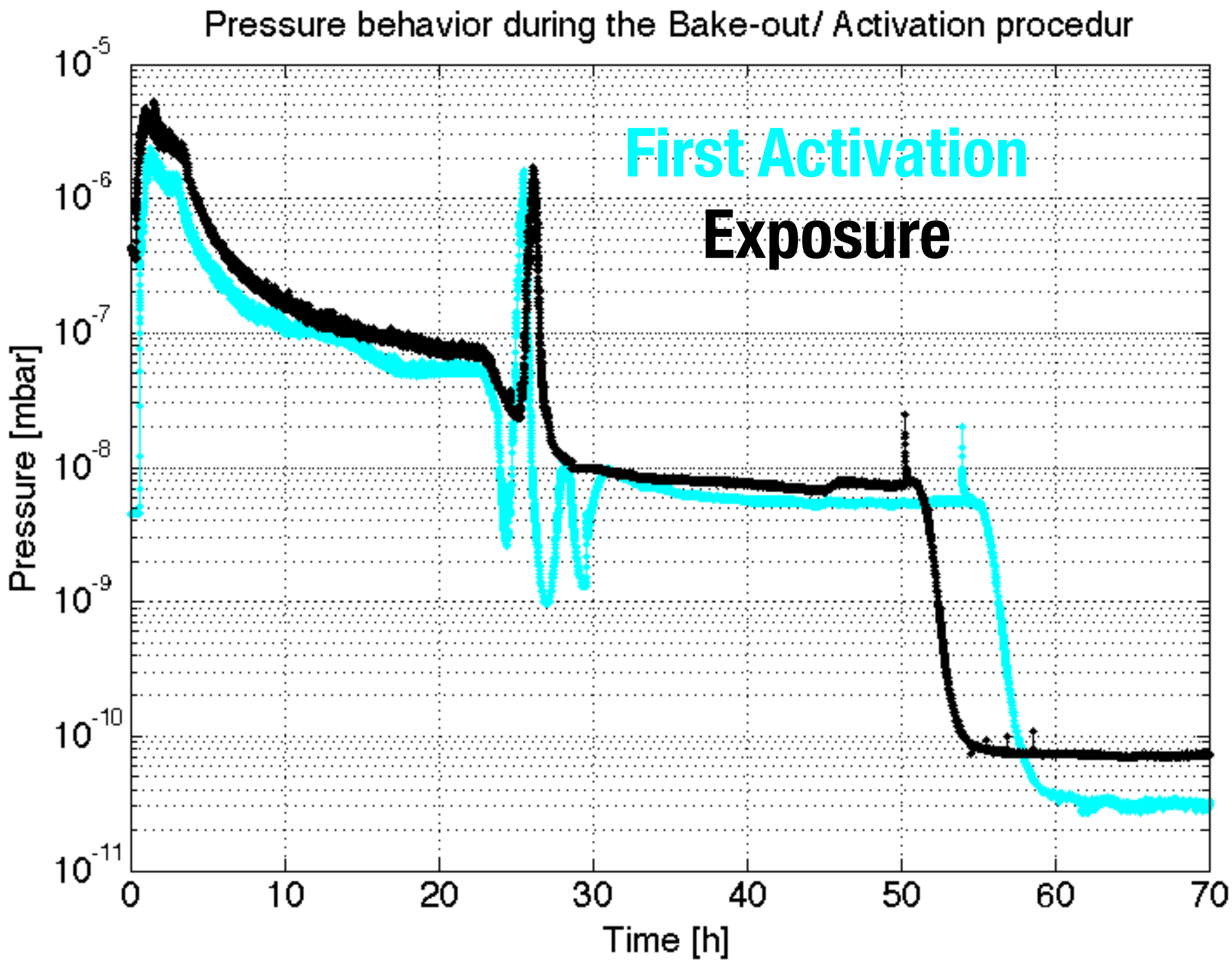
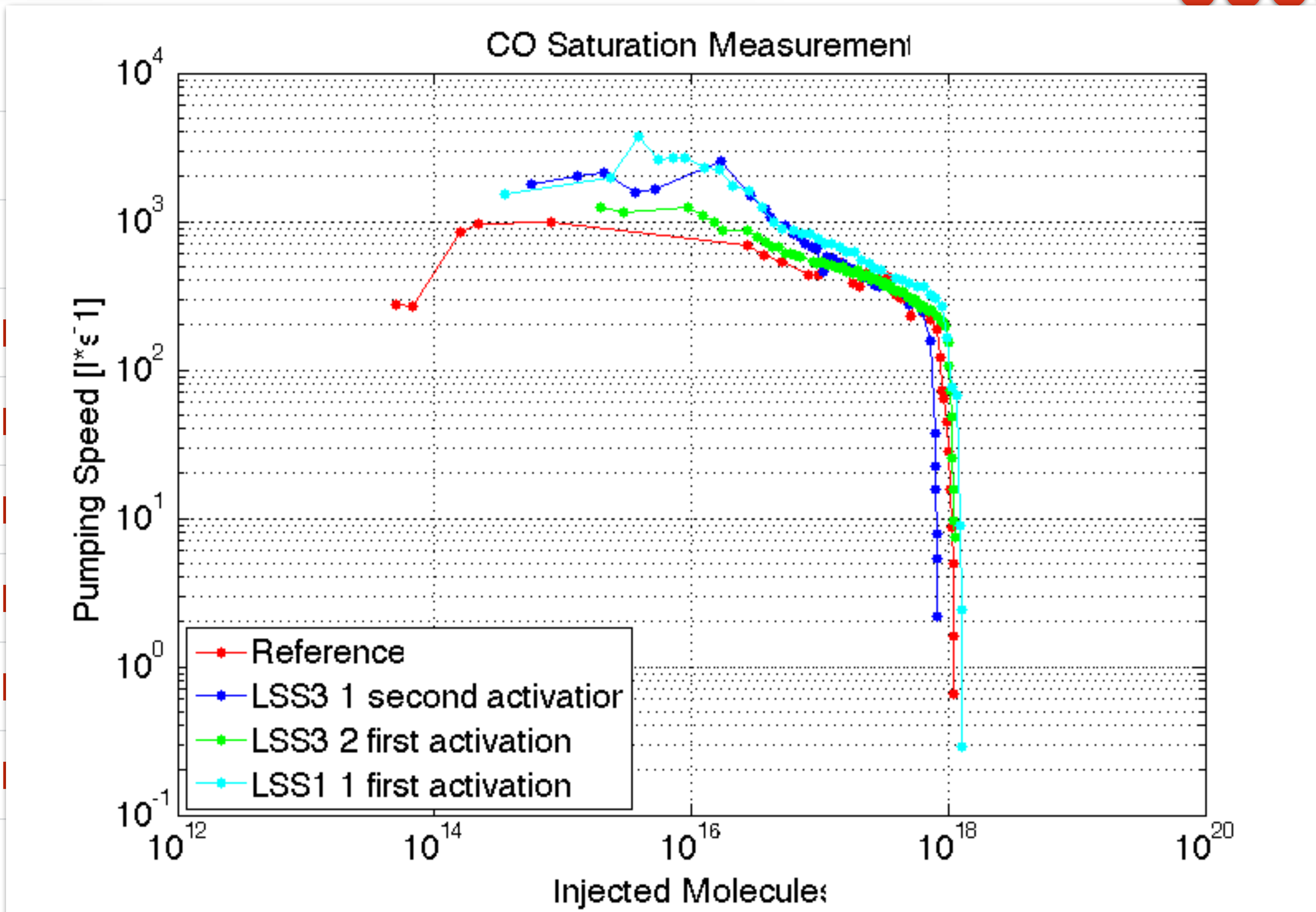


Experimental Results

Ageing Campaign

SAMPLE	CO Molecules		
	I Act	II Act	Vented
Reference	8 10	-	-
LSS3_1	-	7 10	5 10
LSS3_2	1 10	-	-
LSS1_1	1 10	-	-
LSS1_2	NOT TESTED		
N2 sealed	NOT LEAK TIGHT		

Experimental Results



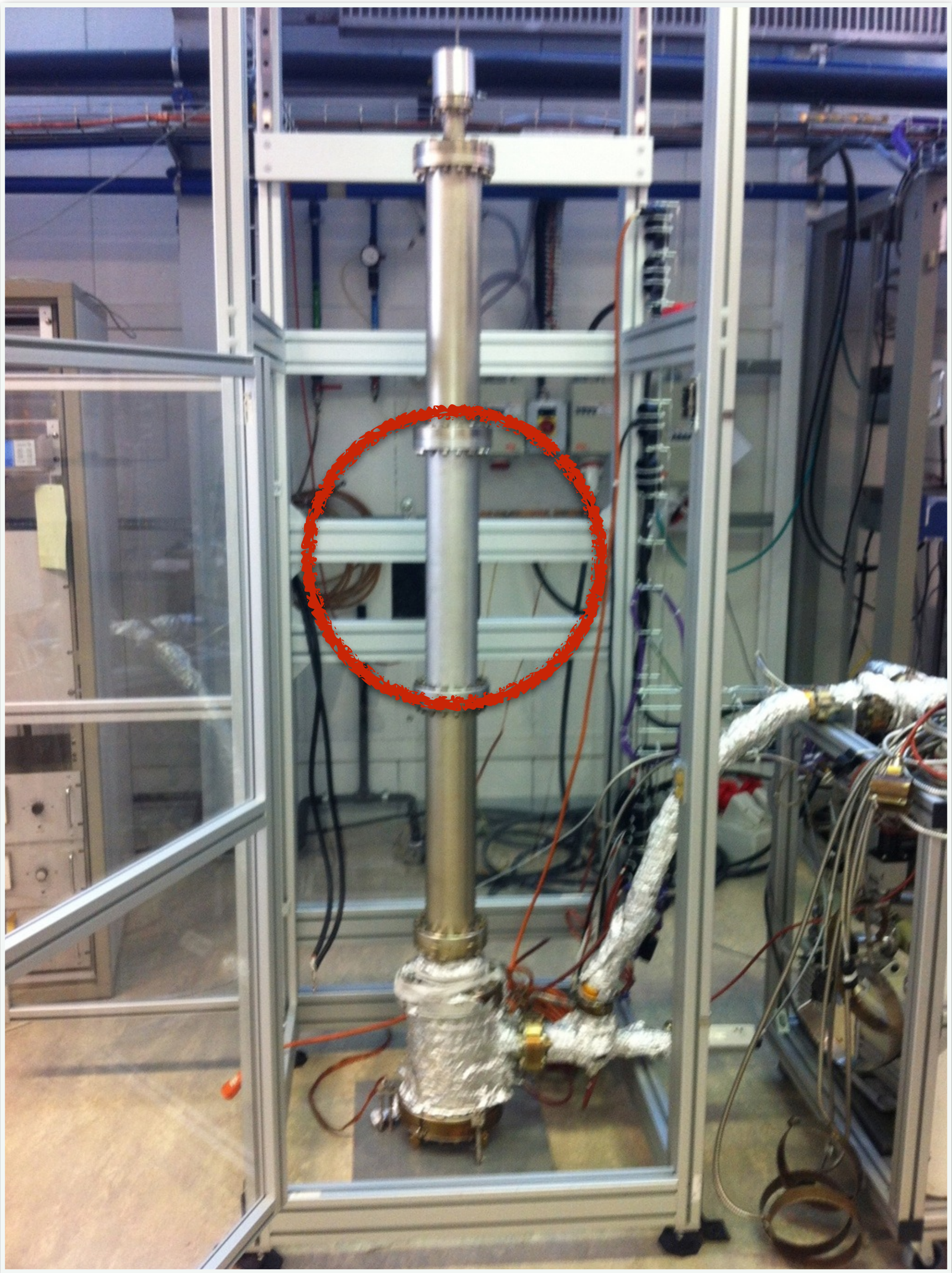
Coating Campaign

Compositions tested

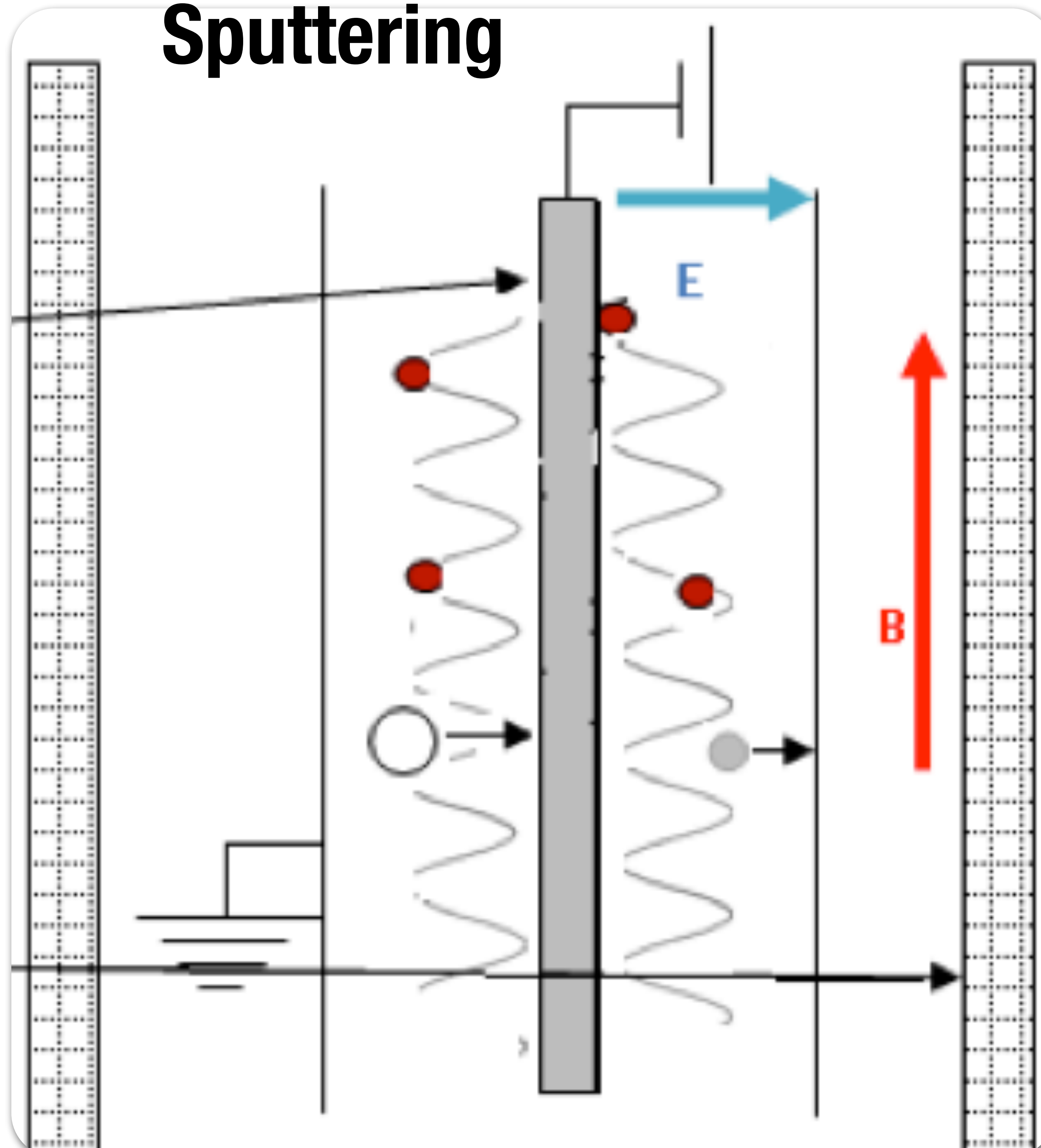
- ZrV
- HfV
- TiZrHfV

Experimental Set-Up

Coating Tower



Magnetron Sputtering



Experimental Set-Up

Coating Campaign

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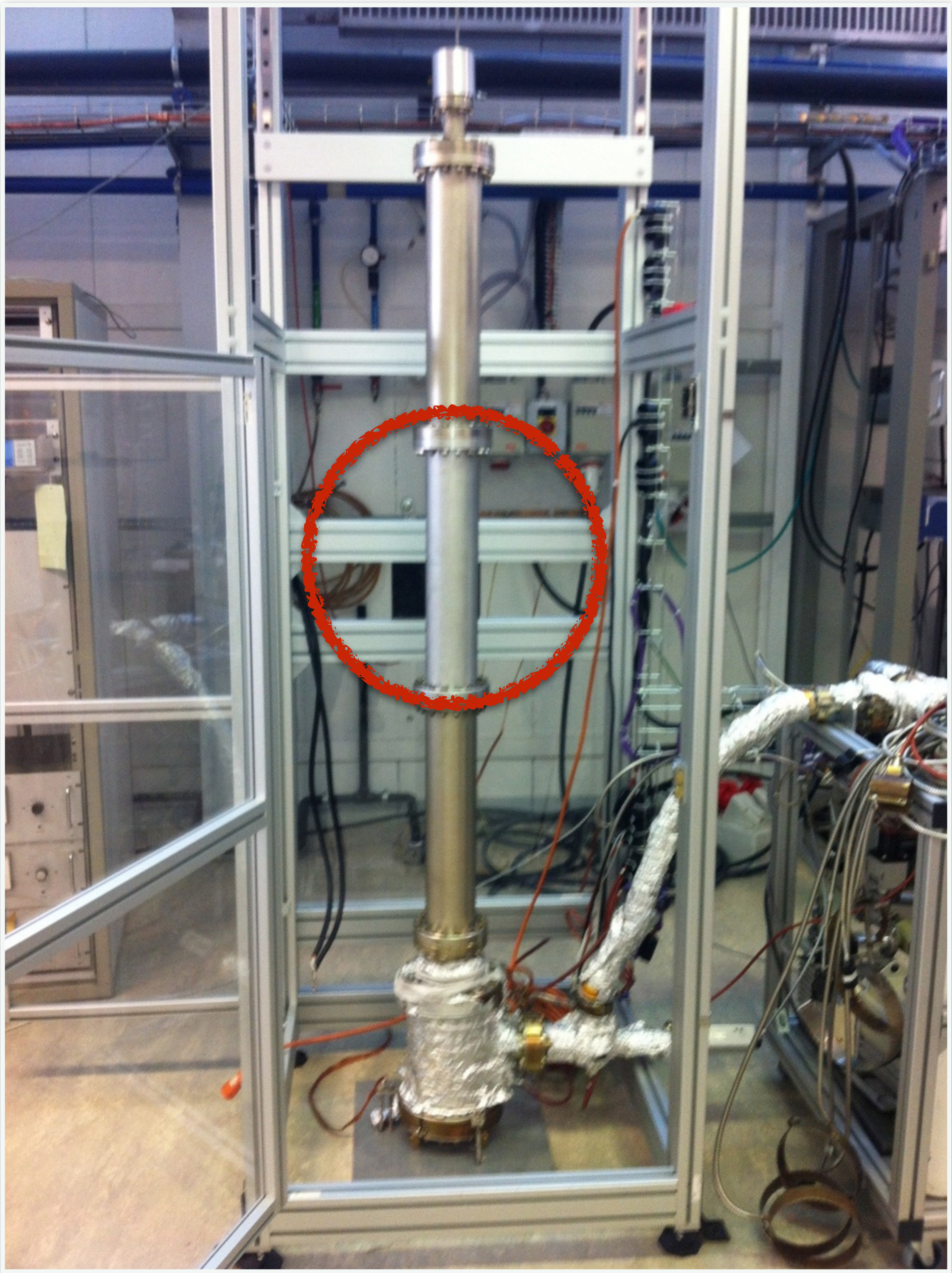

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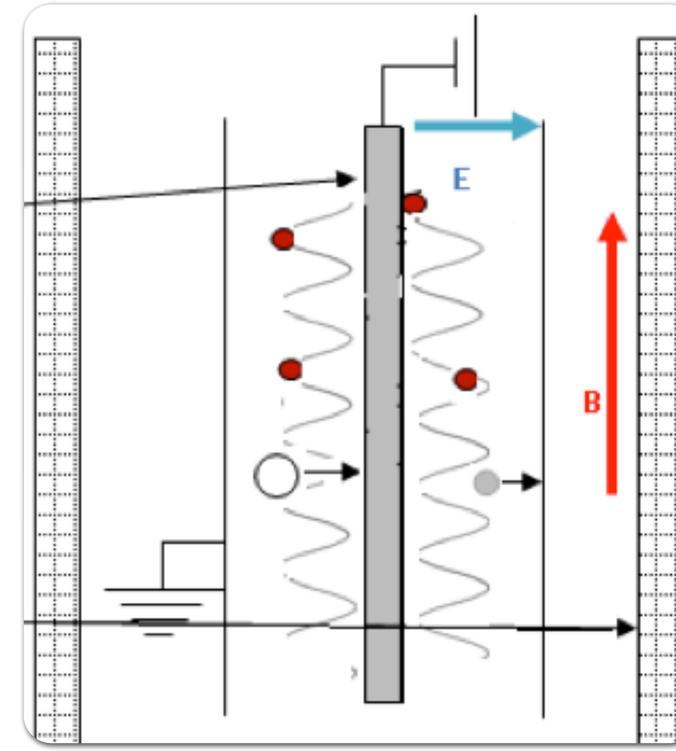
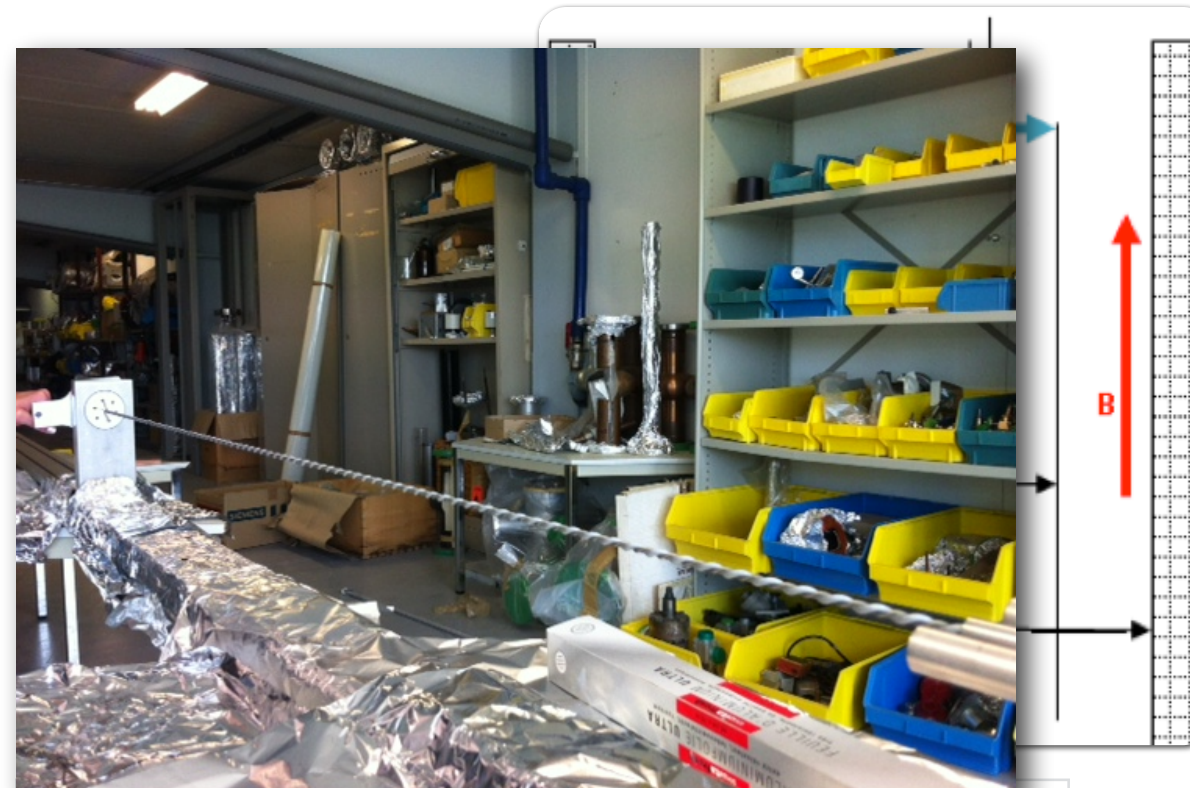


Coating Parameters	
Discharge Gas	kr
Gas Pressure	4,5 10-3 Torr
Solenoid Current	~ 100 A
Solenoid Potential	~ 40 V
Cathode Current	- 600 V
Cathode Potential	0,1 kW (DC)
Cathode Power	0,2 A
Coating Tower Temperature	100 °C

Experimental Set-Up

Coating Tower



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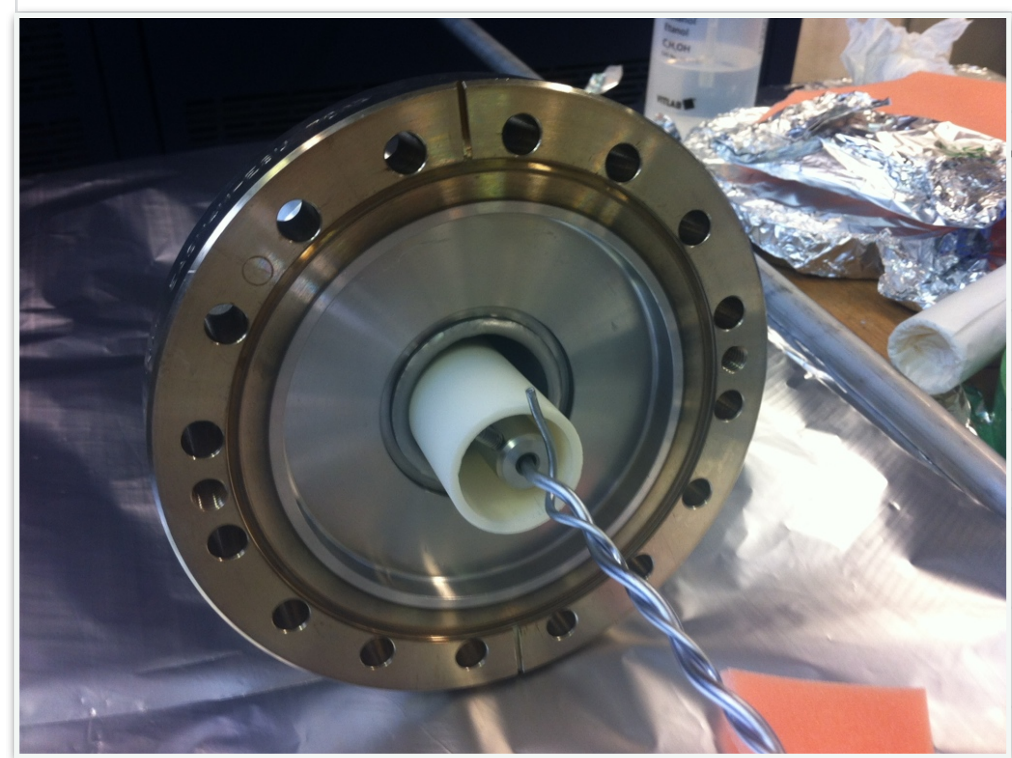


Characterization Measurements

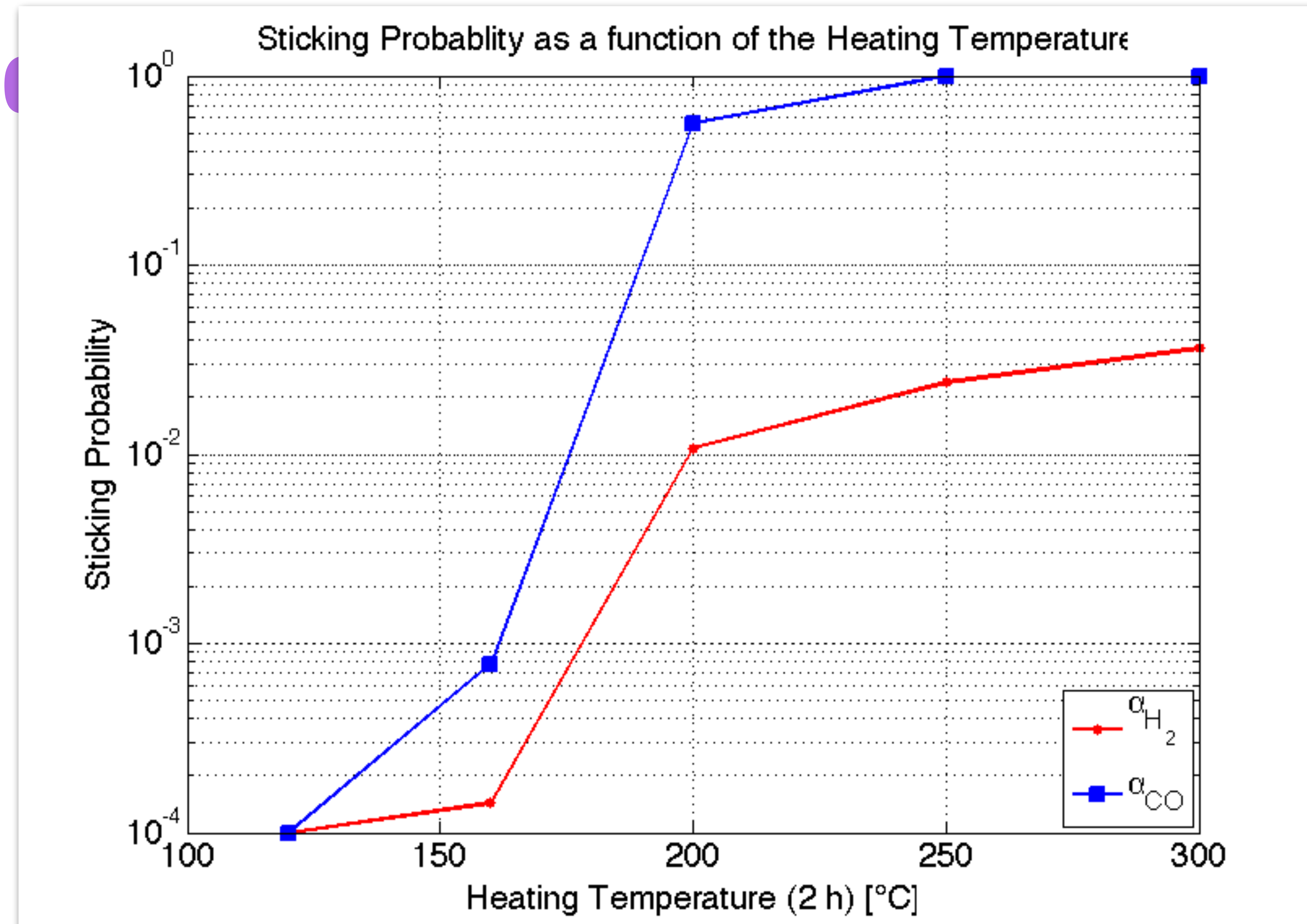
Macroscopic Measurement

- Sticking Probability for H_2 and CO

Characterization Measurements

Macroscopic Measurement

- Sti



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Characterization Measurements

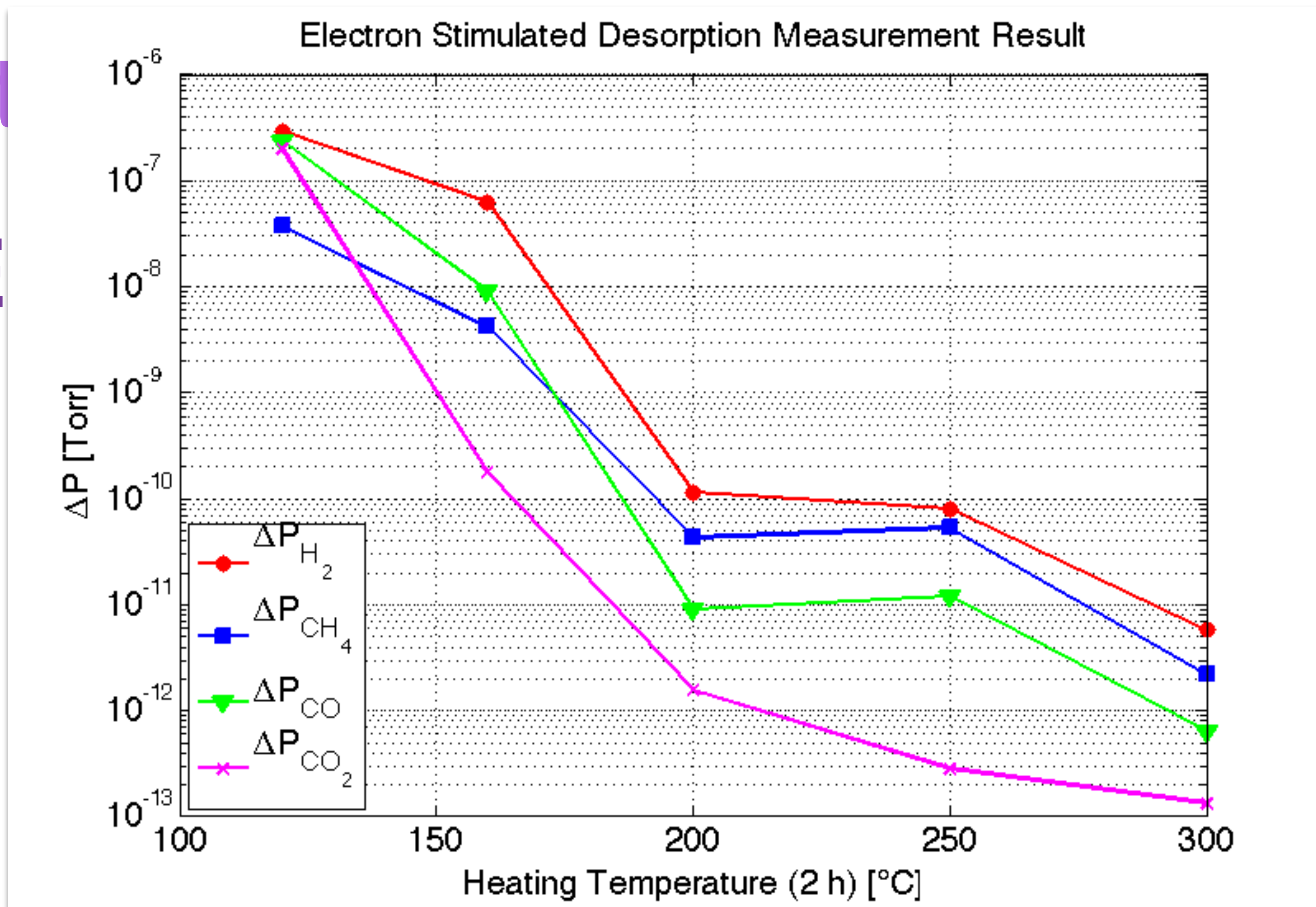
Macroscopic Measurement

- Sticking Probability for H2 and CO
- Electron Stimulated Desorption - ESD

Characterization Measurements

Macroscopic Measurement

- St
- E ... D



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Macroscopic Measurement

- Sticking Probability for H_2 and CO
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Characterization Measurements

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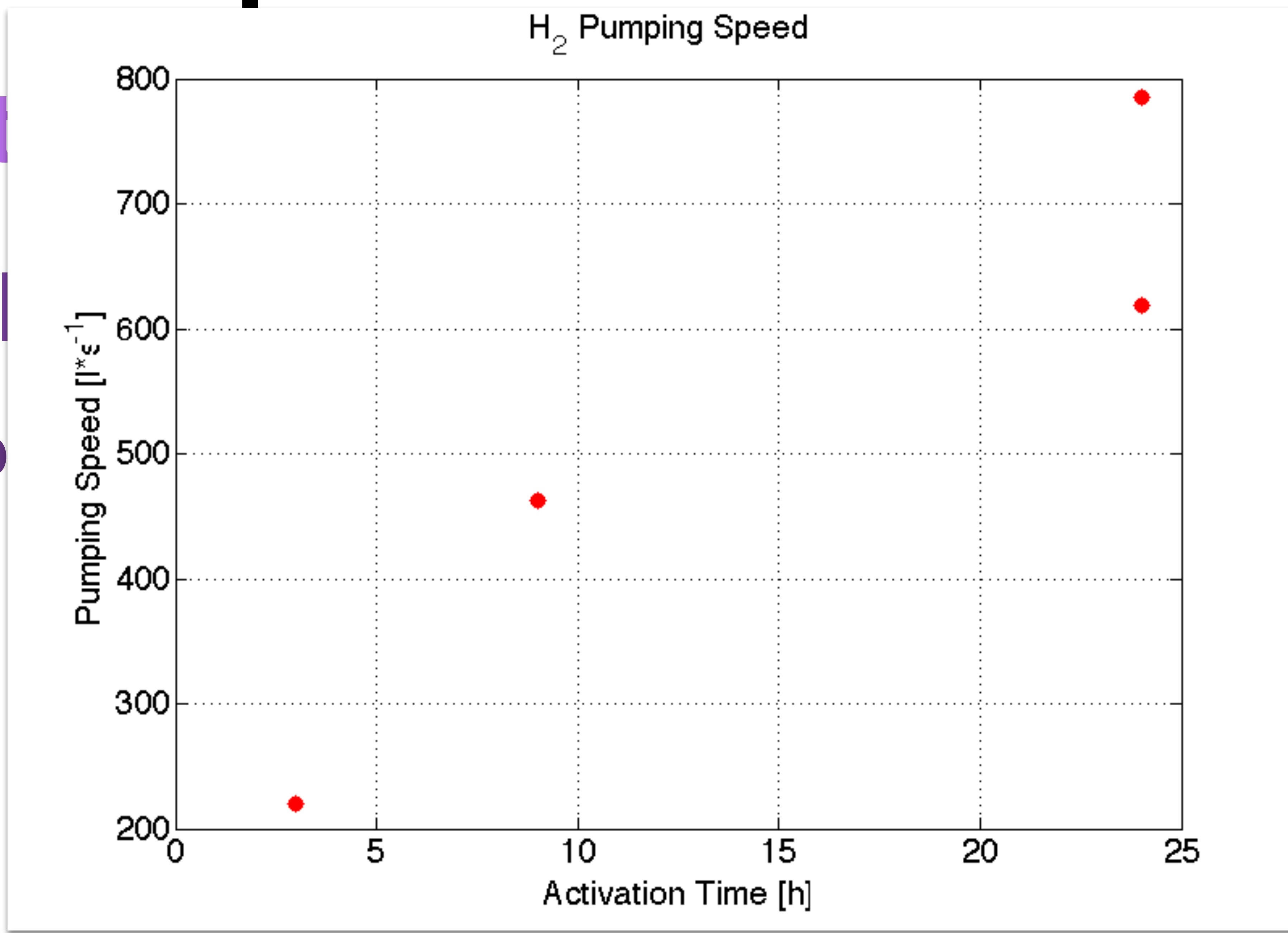
- Pumping Speed Measurement

Characterization Measurements

Macroscopic Measurement

- St
- El D
- P



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- Pumping Speed Measurement

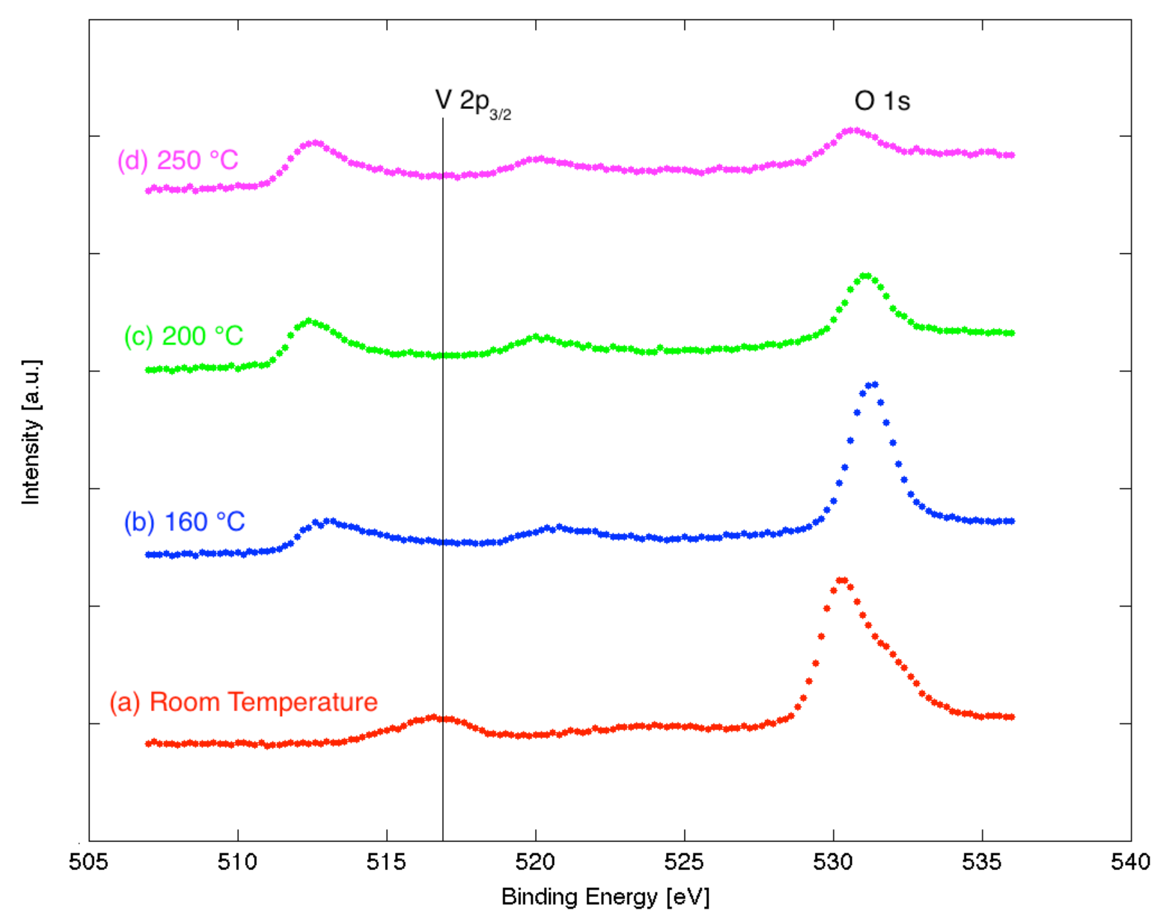
Characterization Measurements

Microscopic Measurement

Characterization Measurements

Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS



Characterization Measurements

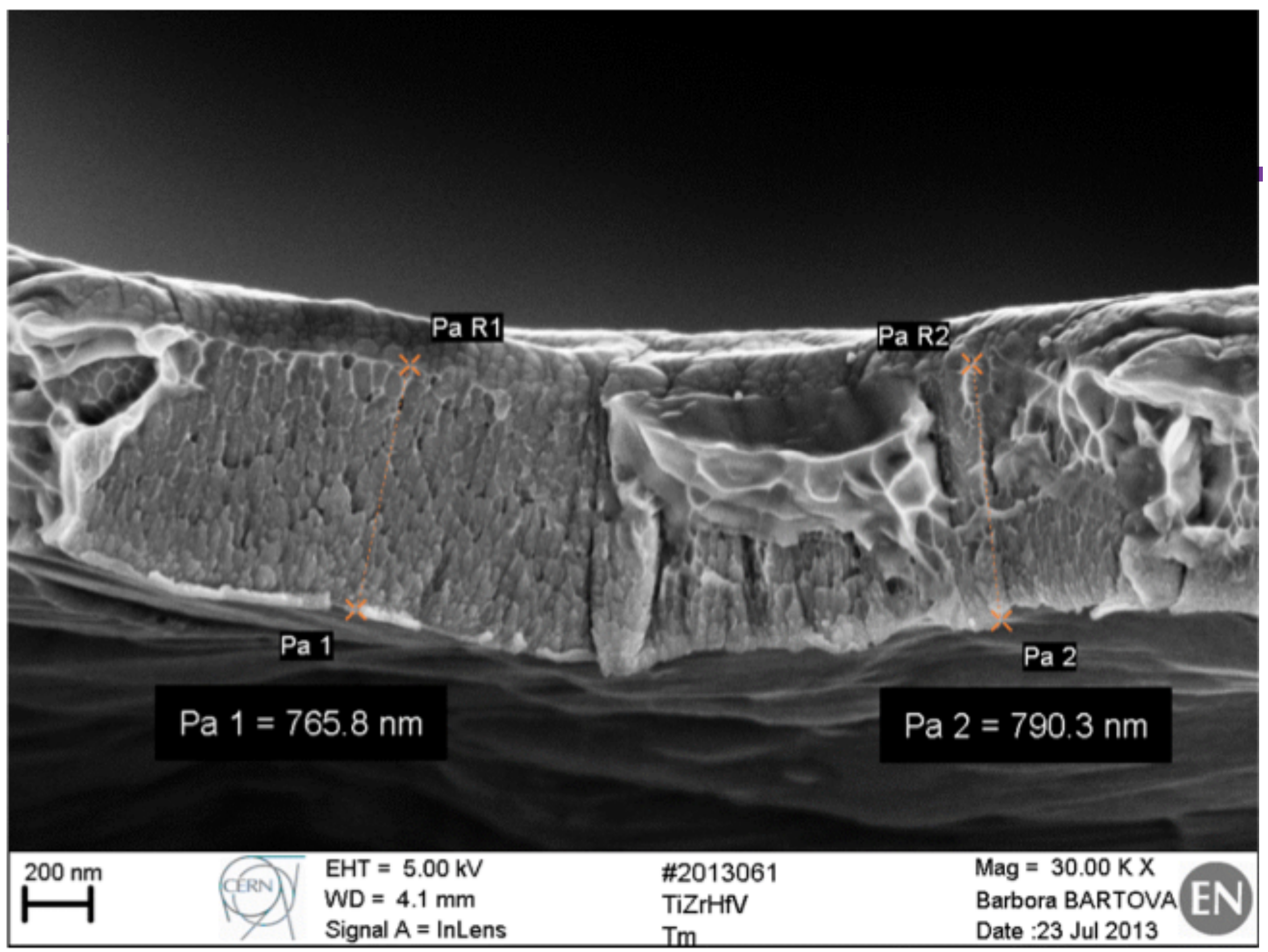
Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS
- Scanning Electron Microscopy - SEM

Characterization Measurements

Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS
- Scanni - SEM



Characterization Measurements

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Characterization Measurements

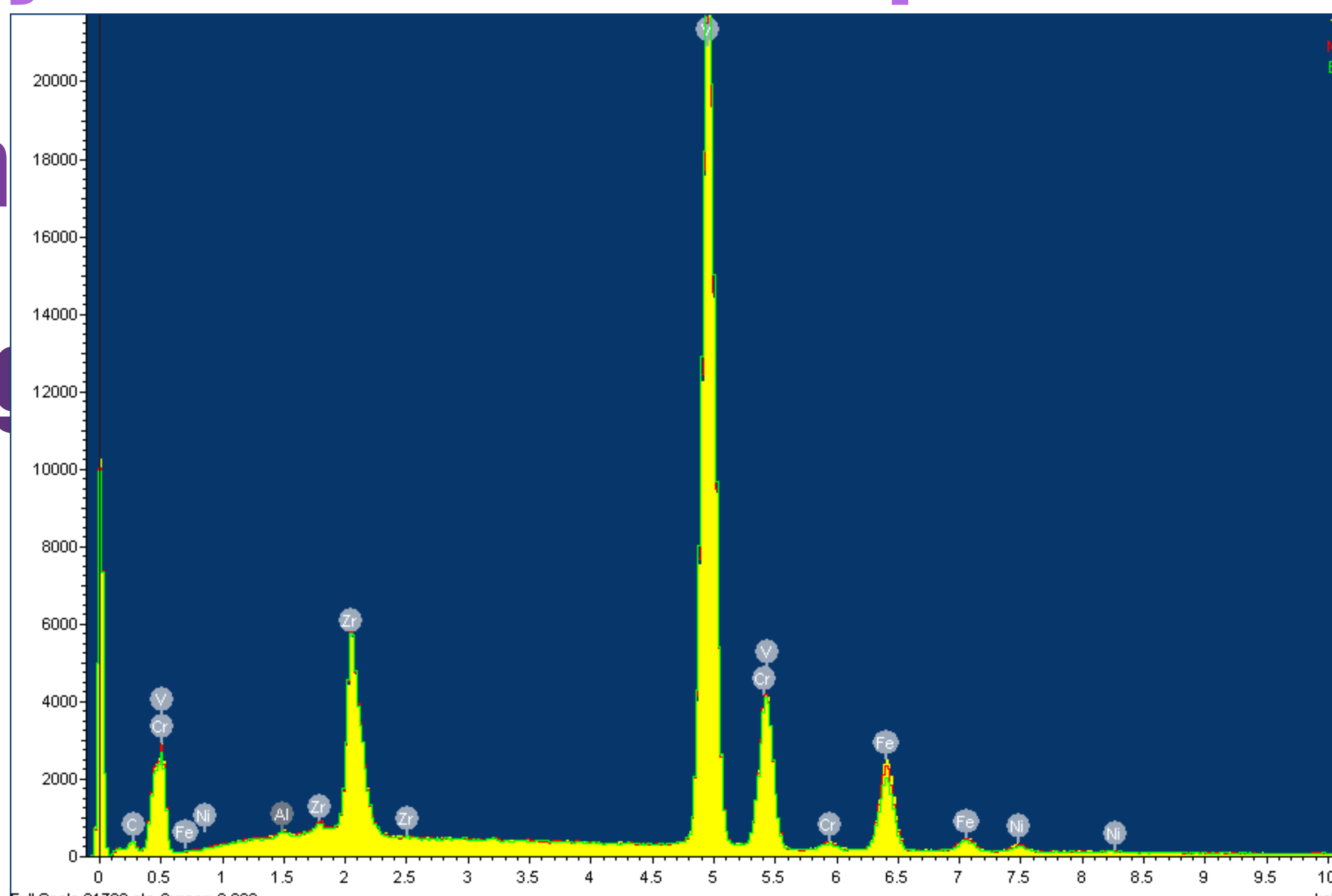
Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS
- Scanning Electron Microscopy - SEM
- Energy Dispersive Spectroscopy - EDS

Characterization Measurements

Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS
- Scan … SEM
- Energ … - EDS



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Characterization Measurements

Microscopic Measurement

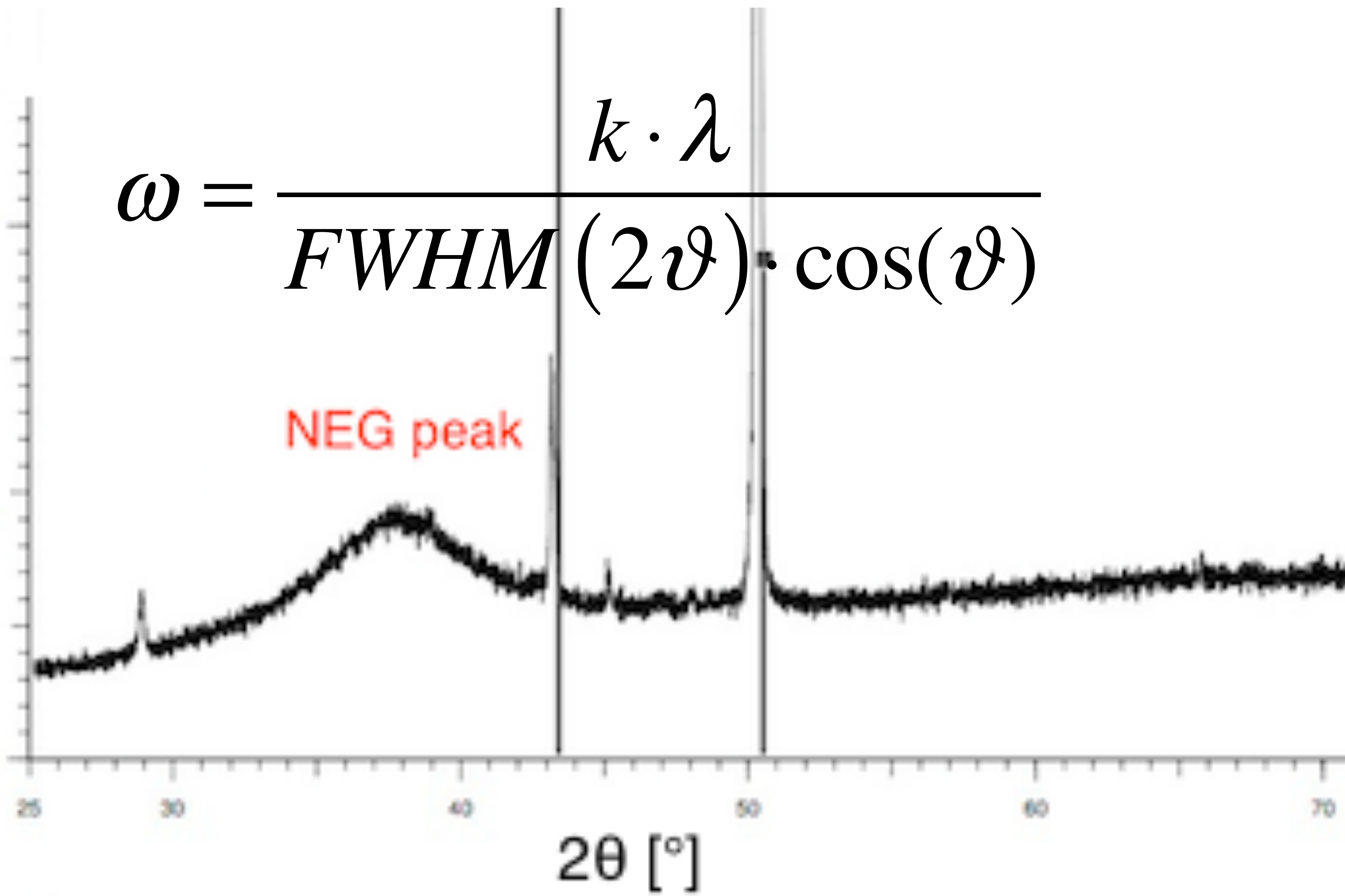
- X-Rays Photoelectron Spectroscopy - XPS
- Scanning Electron Microscopy - SEM
- Energy Dispersive Spectroscopy - EDS
- X-Rays Diffraction Measurement -XRD

Characterization Measurements

Microscopic Measurement

- X · XPS
- S
- E S
- ⟩ RD

$$\omega = \frac{k \cdot \lambda}{FWHM(2\vartheta) \cdot \cos(\vartheta)}$$



Coating Campaign - ZrV

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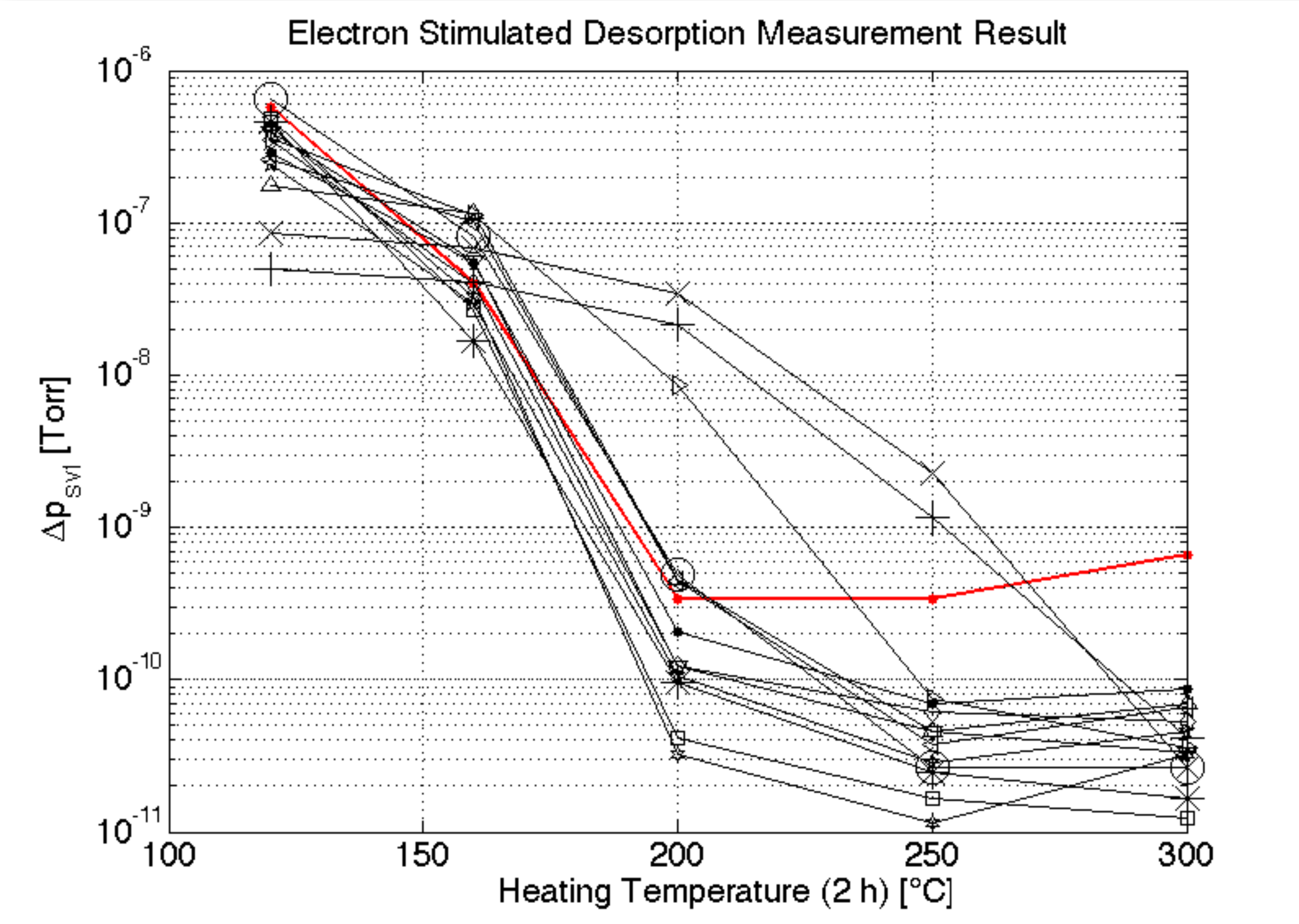
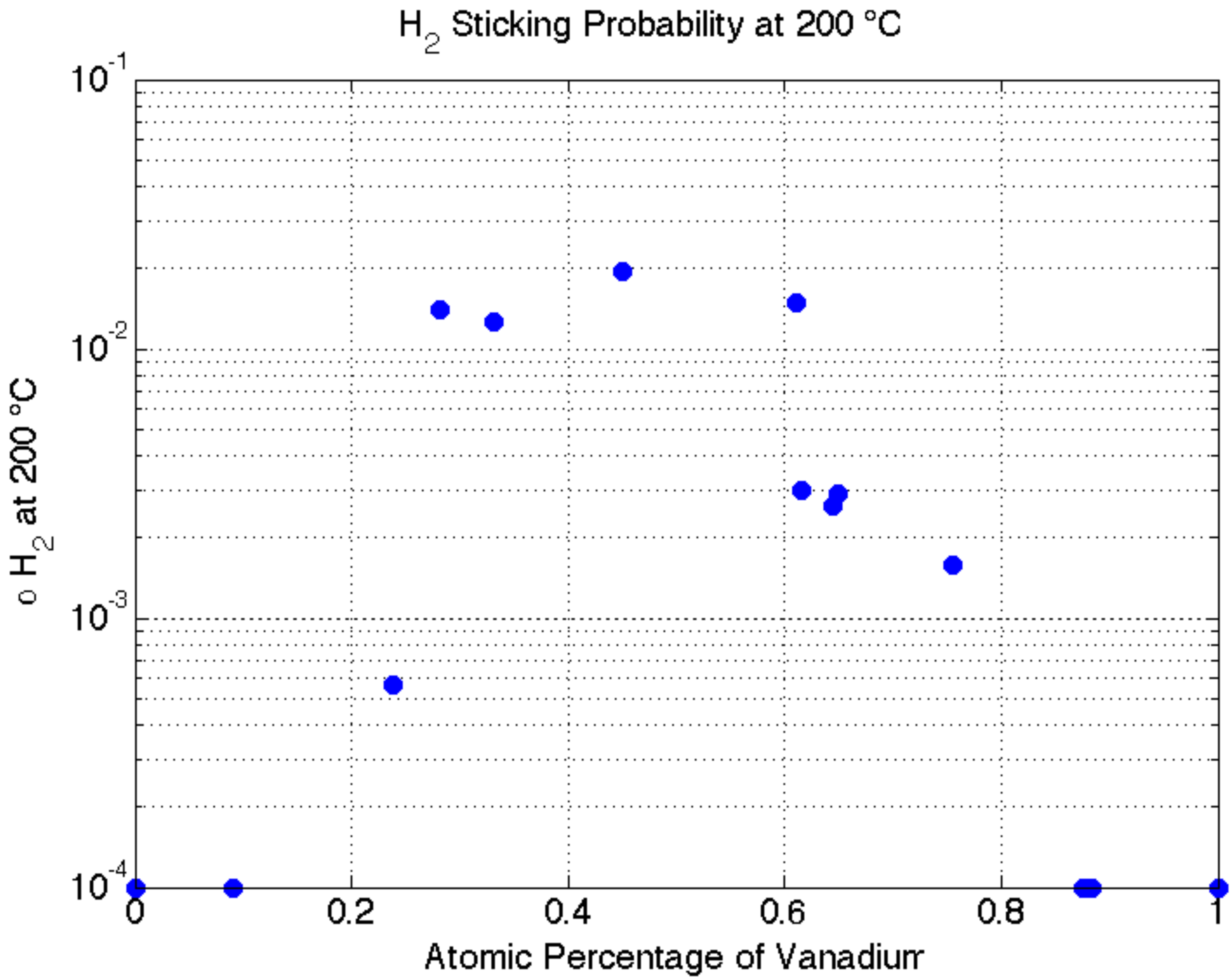
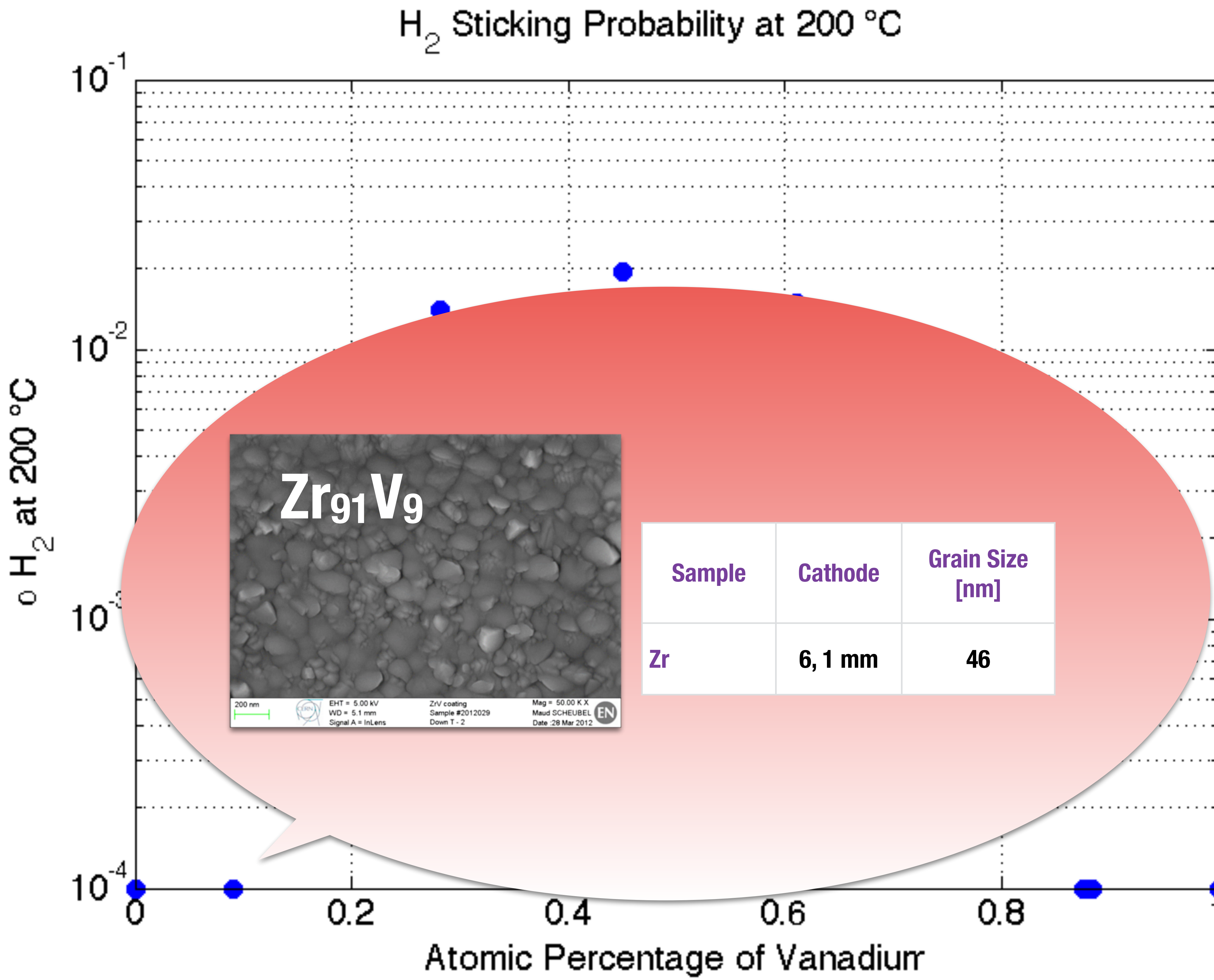


Coating Campaign - ZrV

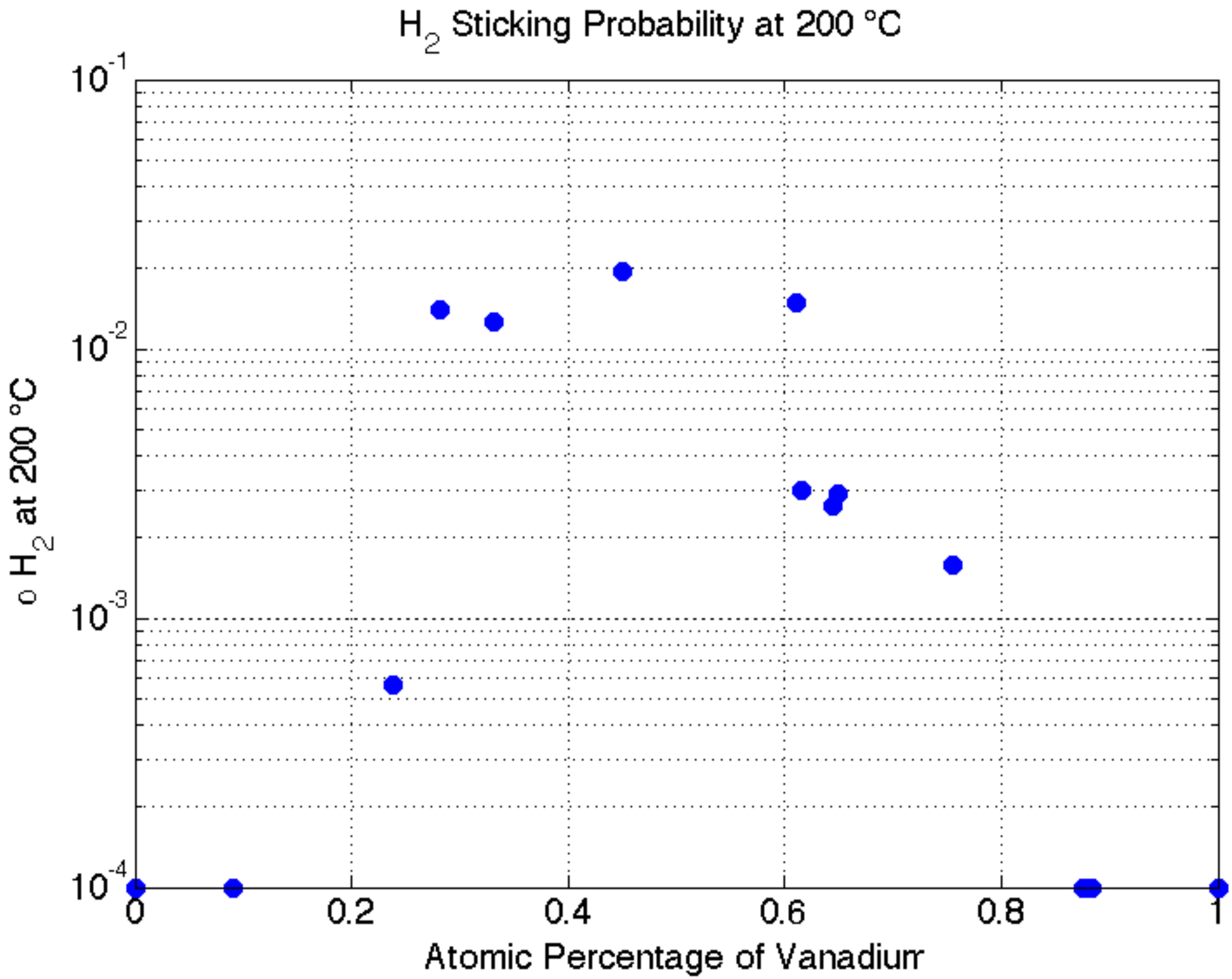


Coating Campaign - ZrV

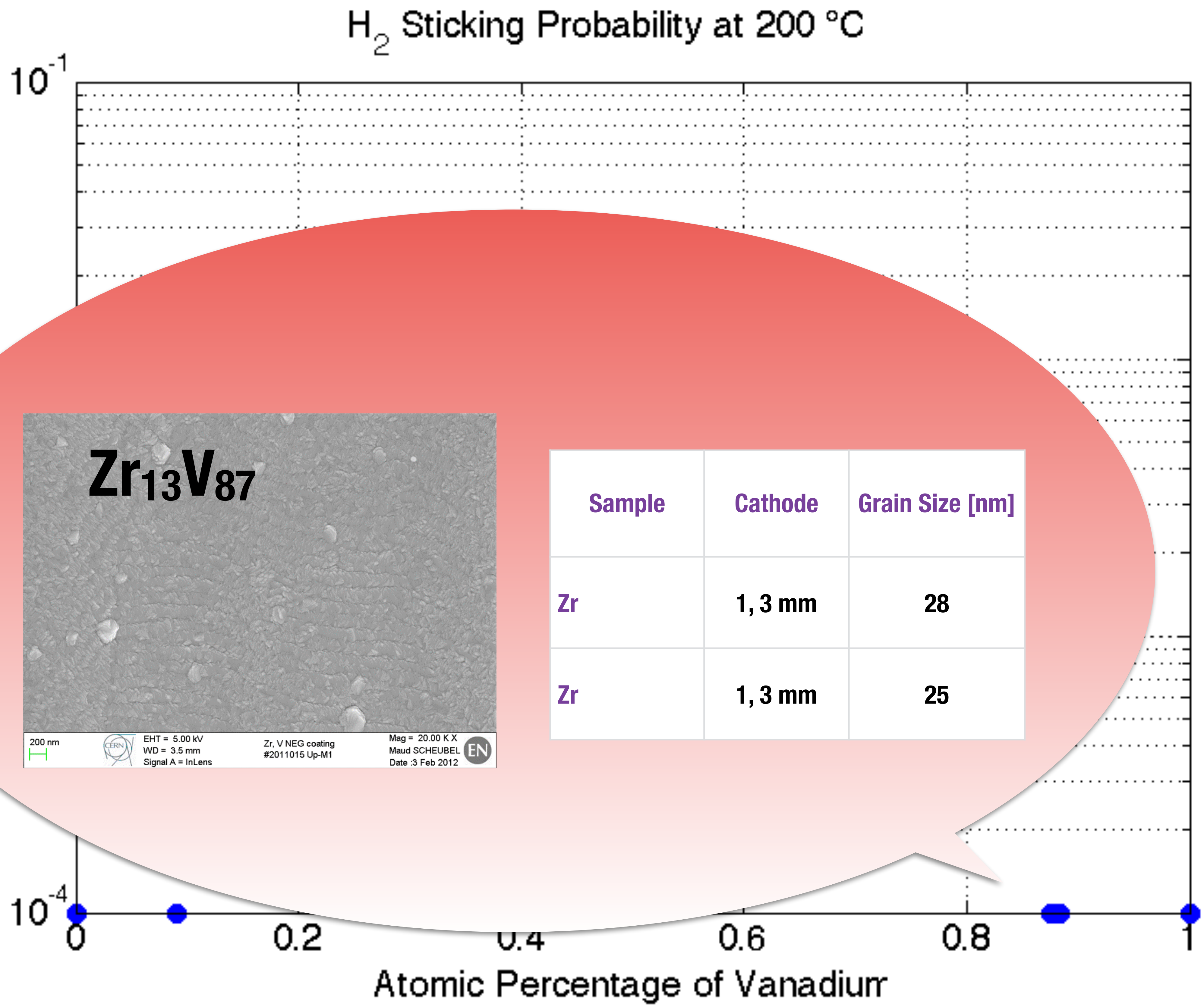


Sample	Cathode	Grain Size [nm]
Zr	6, 1 mm	46

Coating Campaign - ZrV

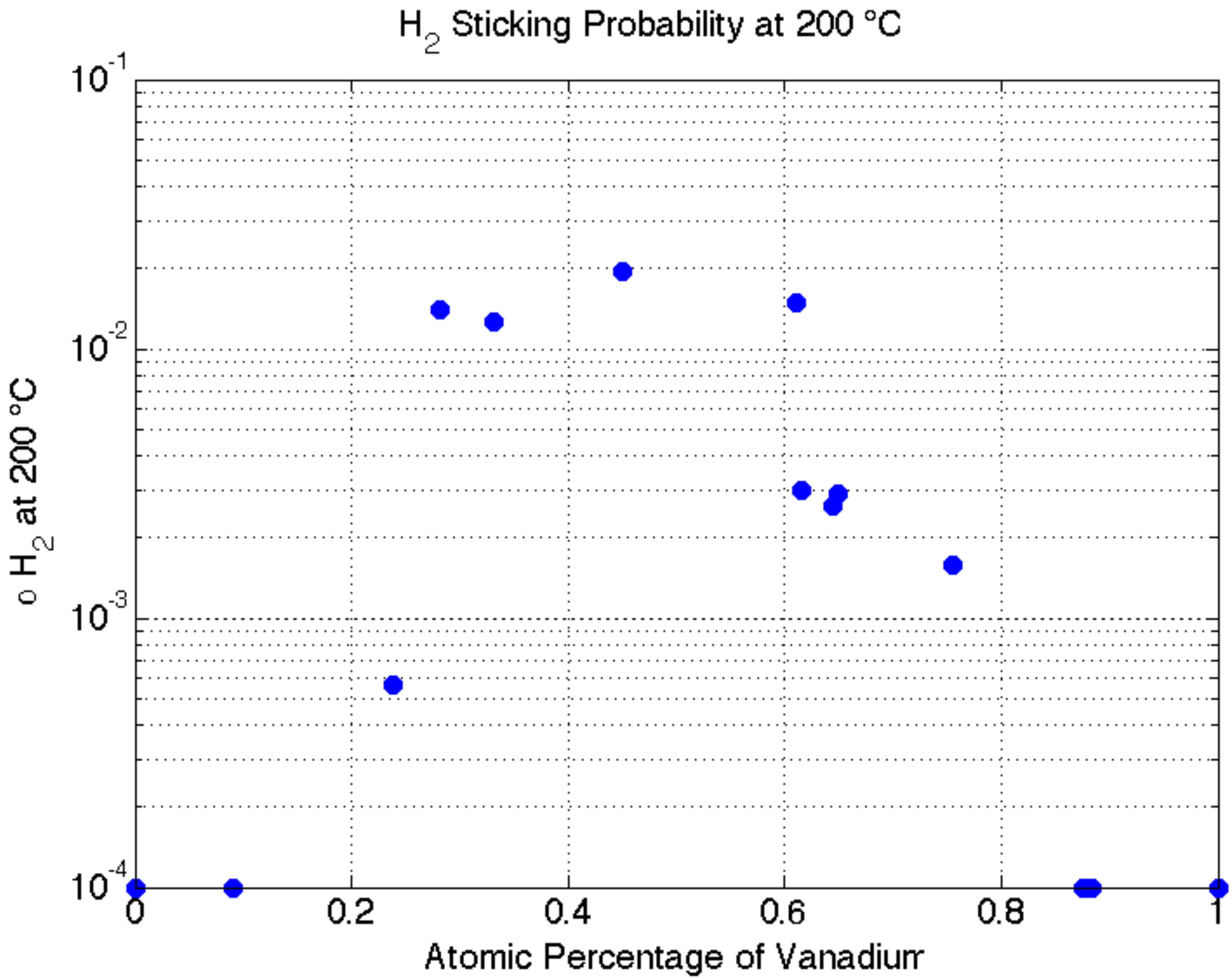


Coating Campaign - ZrV

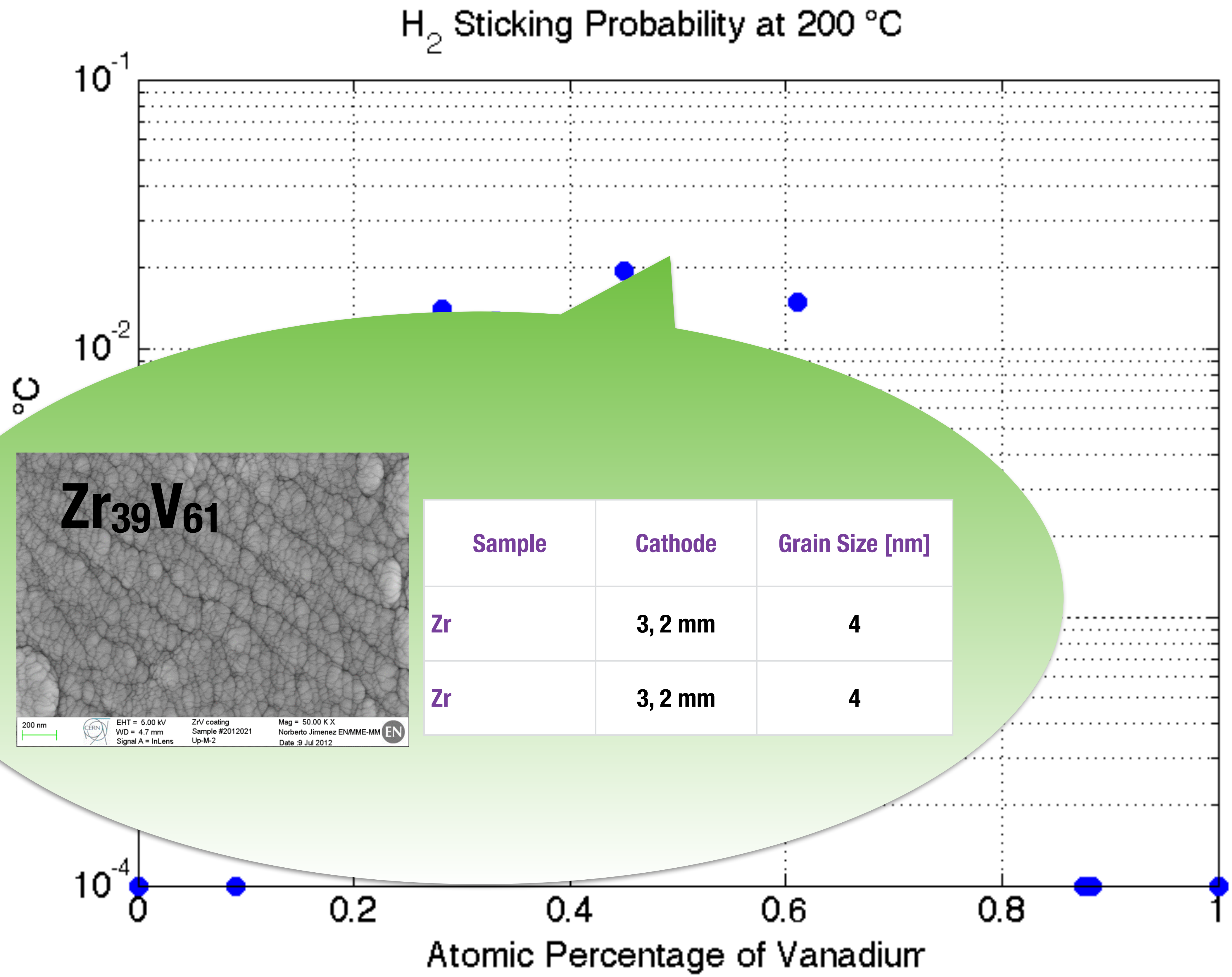


Sample	Cathode	Grain Size [nm]
Zr	1, 3 mm	28
Zr	1, 3 mm	25

Coating Campaign - ZrV



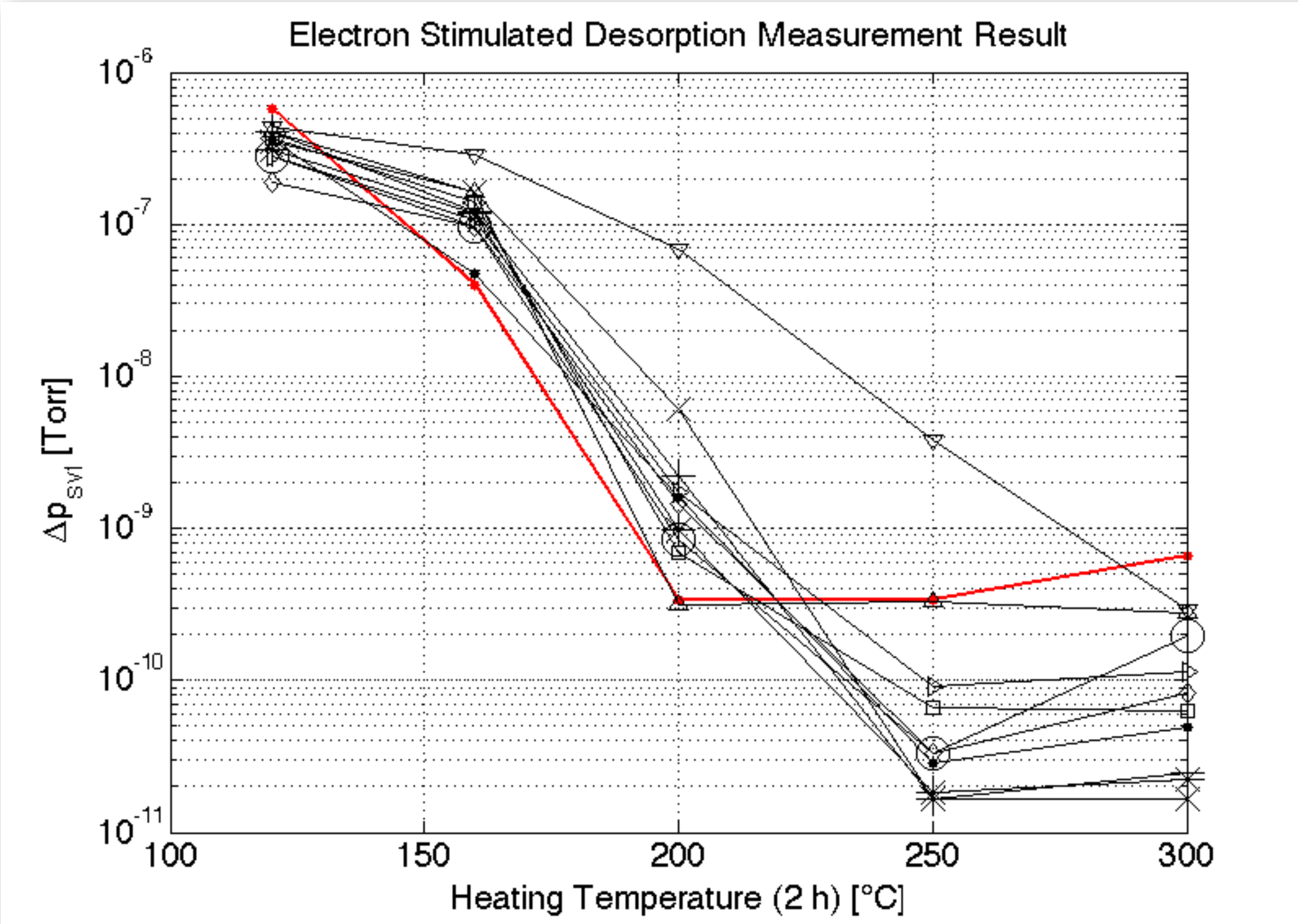
Coating Campaign - ZrV



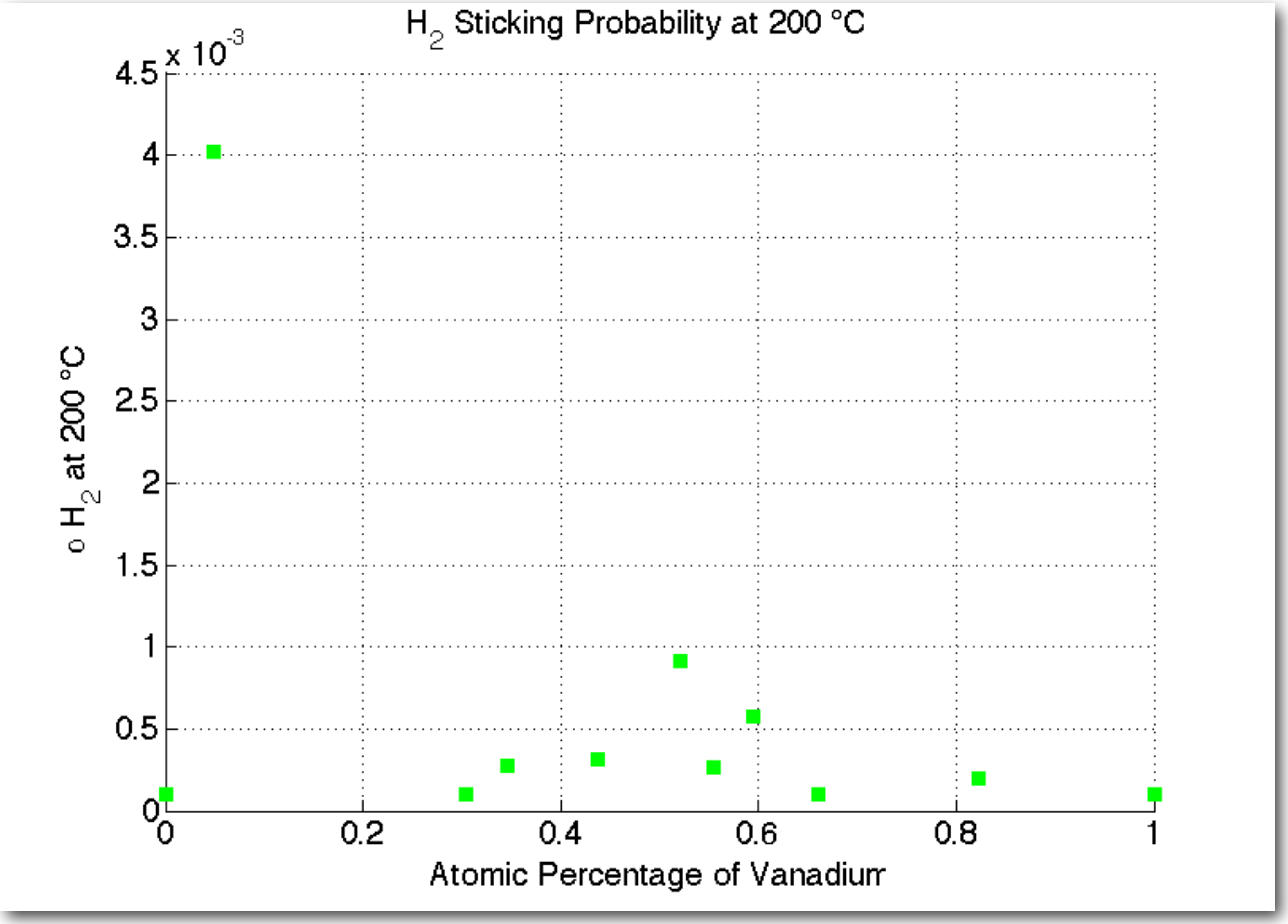
Sample	Cathode	Grain Size [nm]
Zr	3, 2 mm	4
Zr	3, 2 mm	4

Coating Campaign - HfV

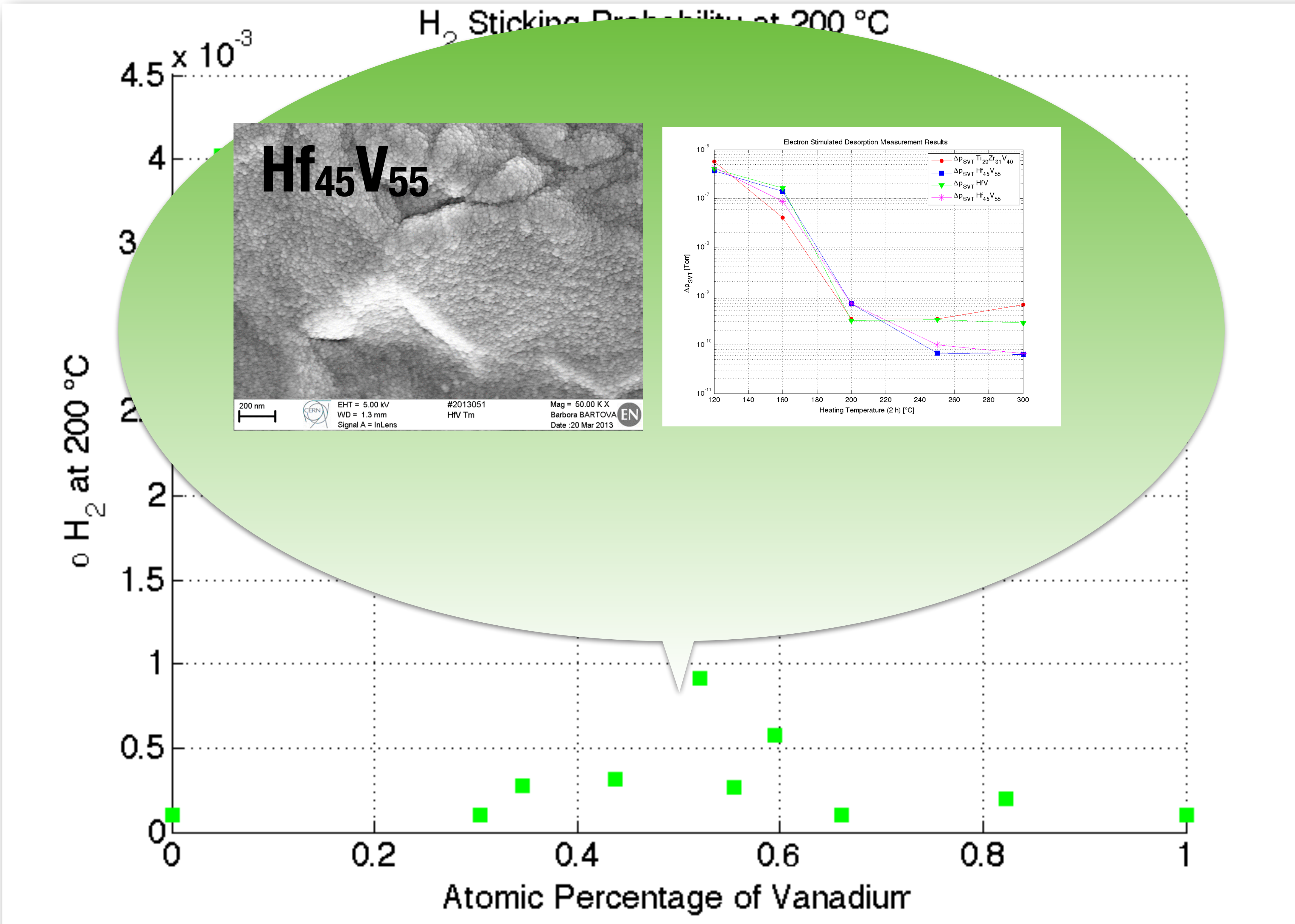
Experimental Results



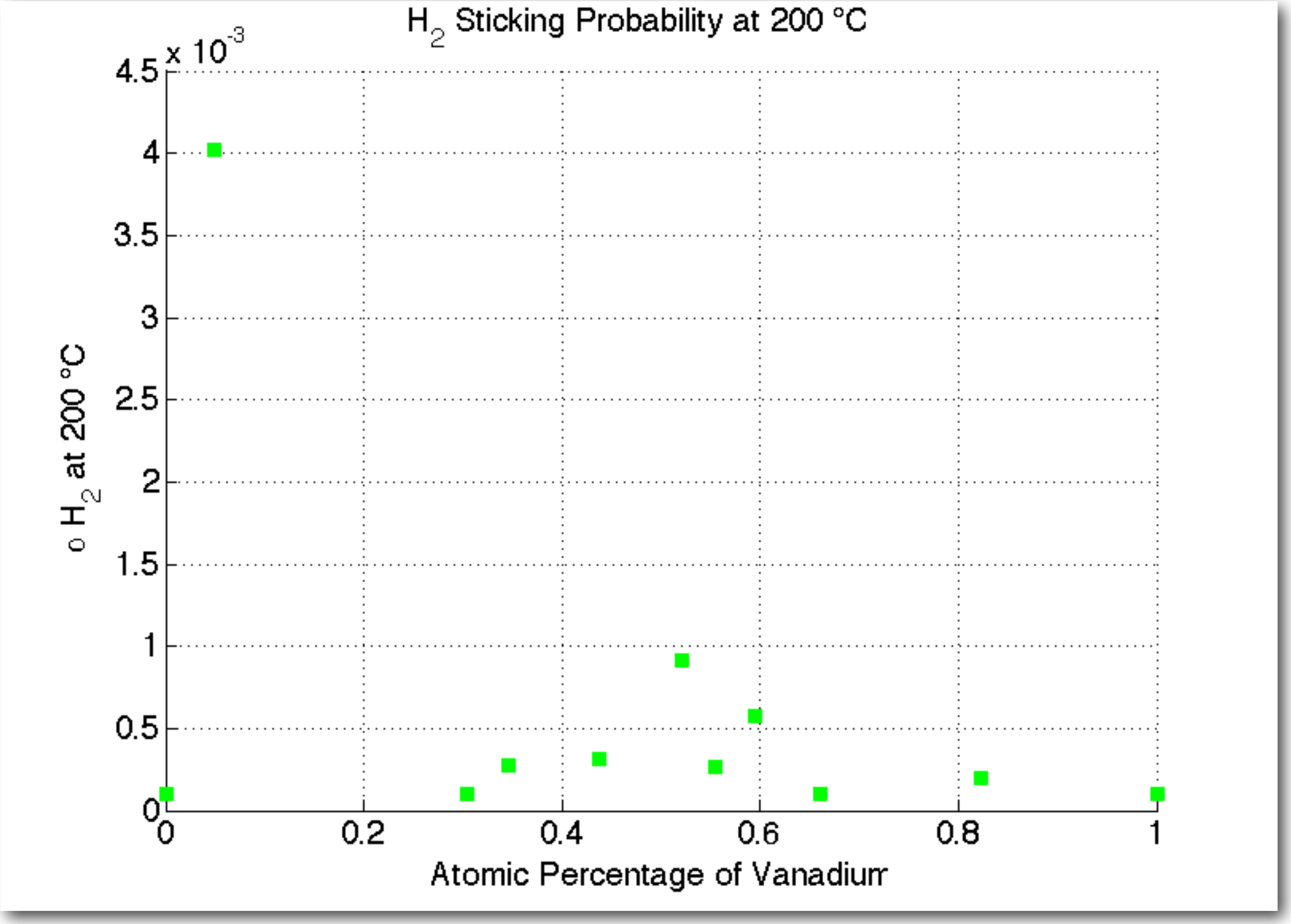
Coating Campaign - HfV



Coating Campaign - HfV

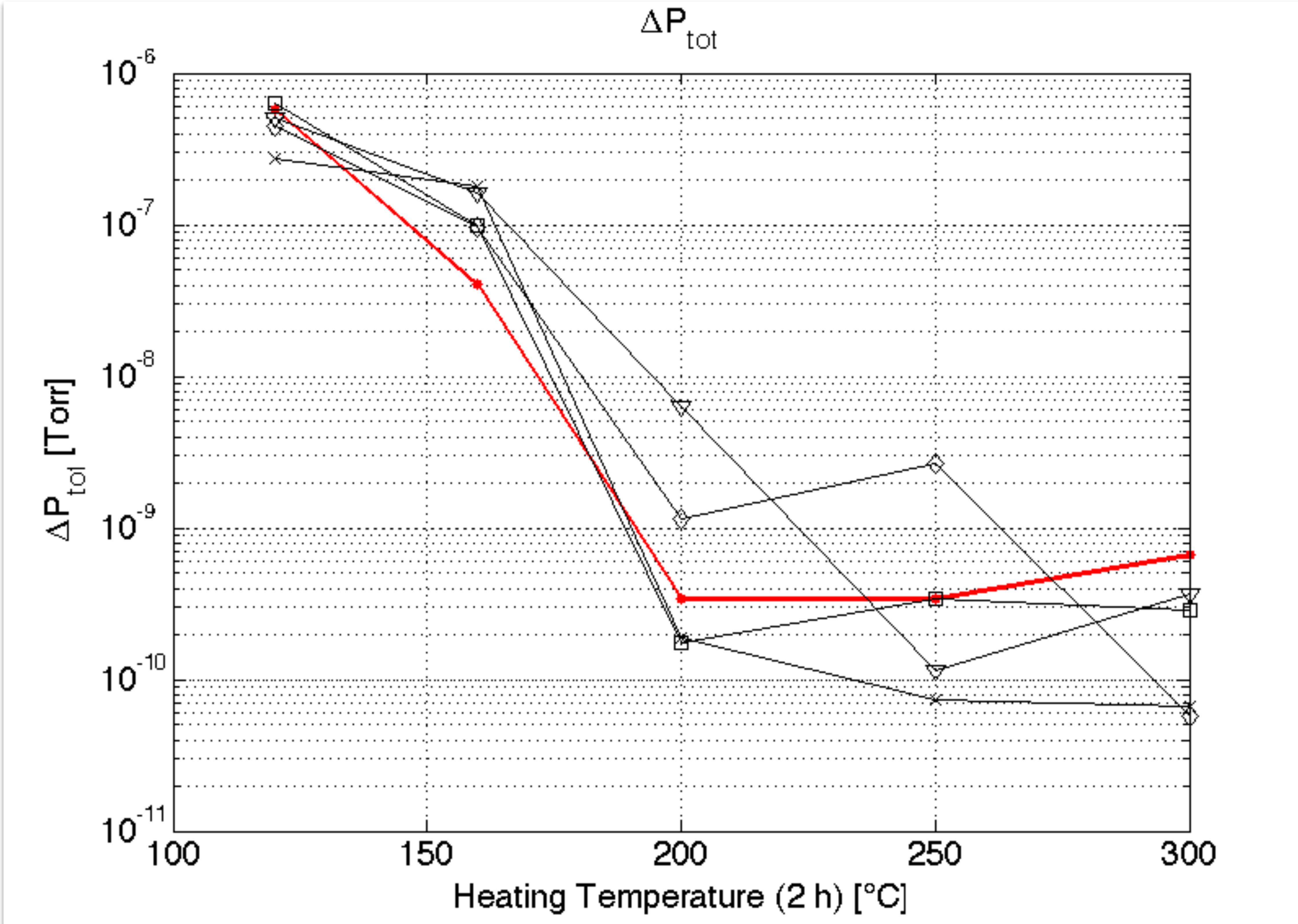


Coating Campaign - HfV

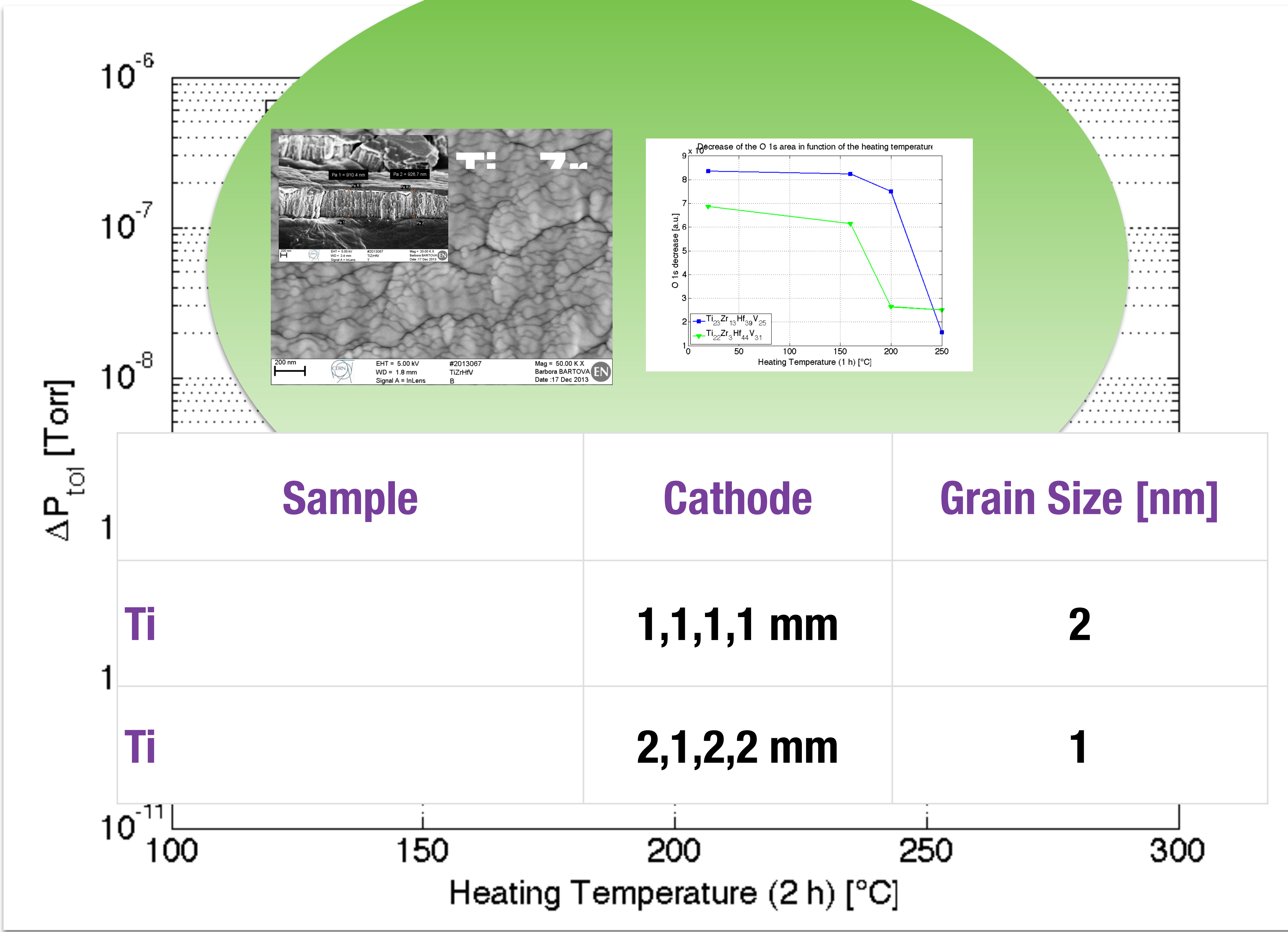


Coating Campaign - TiZrHfV

Coating Campaign - TiZrHfV

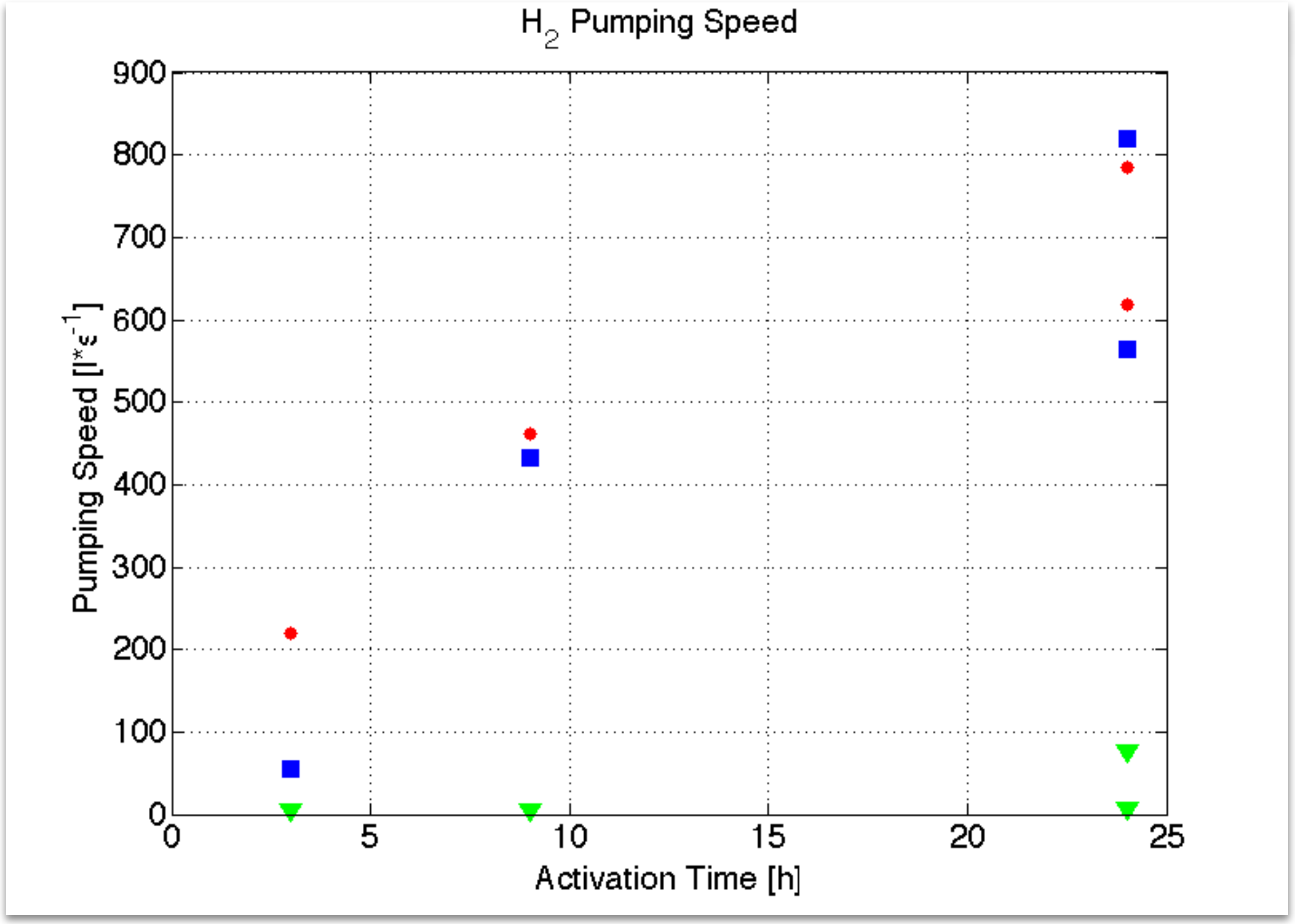


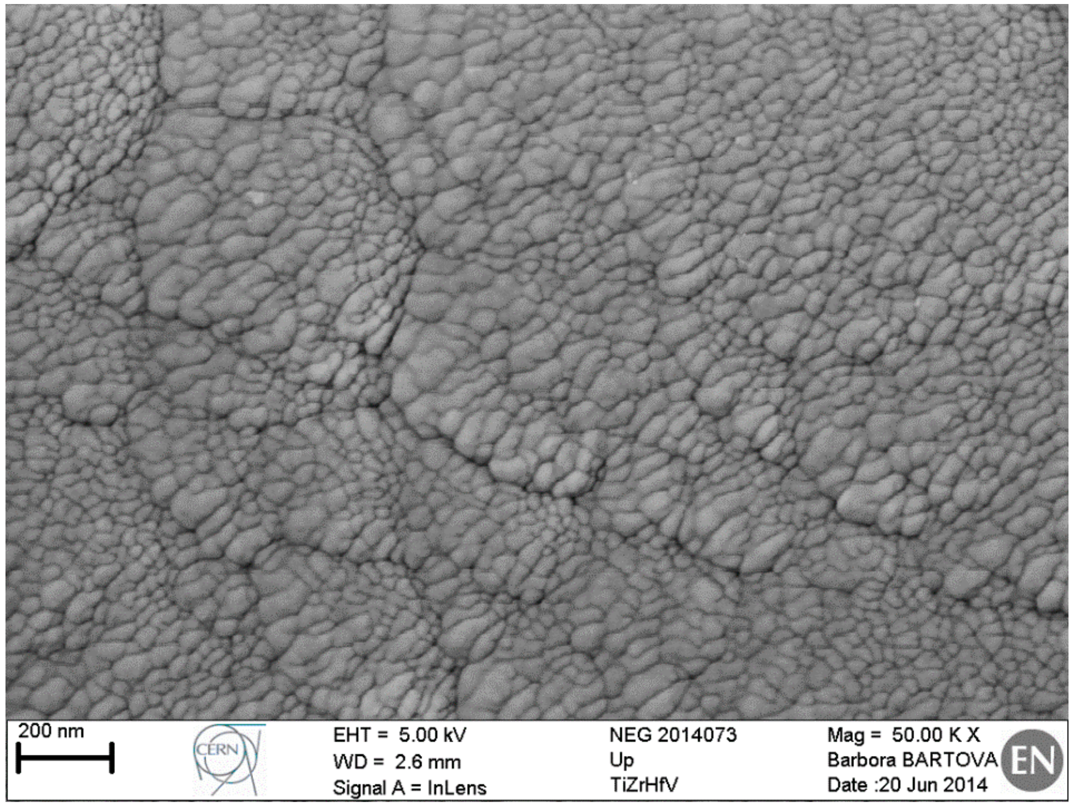
Coating Campaign - TiZrHfV



Sample	Cathode	Grain Size [nm]
Ti	1,1,1,1 mm	2
Ti	2,1,2,2 mm	1

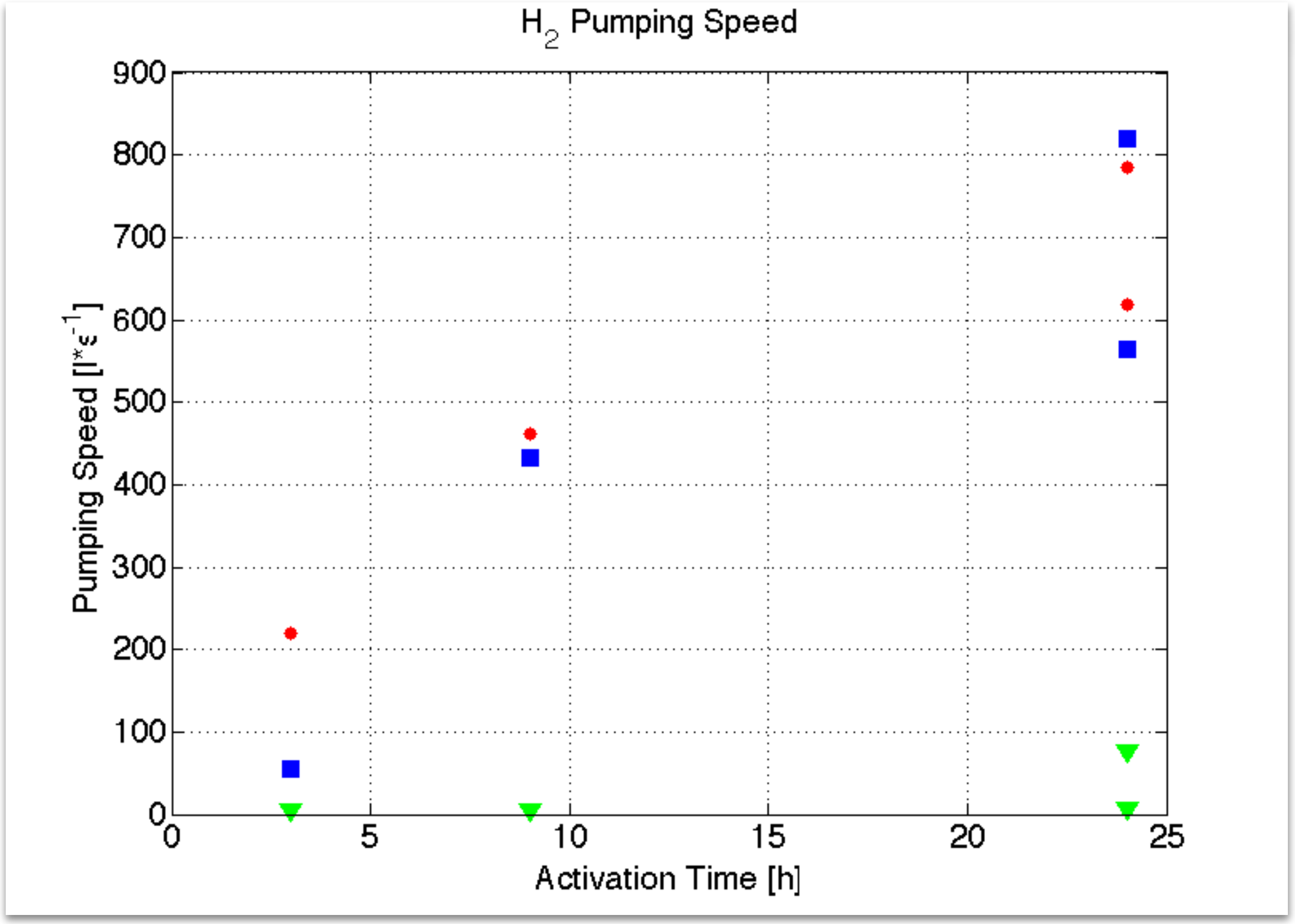
Coating Campaign - TiZrHfV

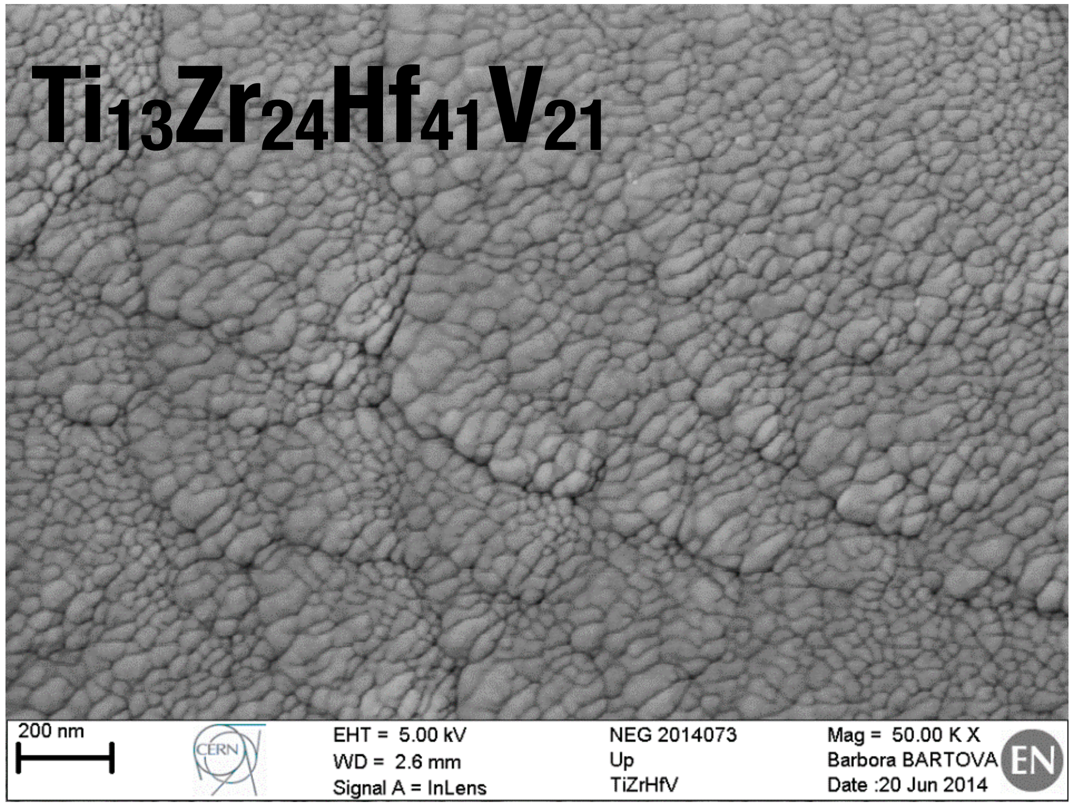




H2 Pumping Speed [l/s]				
SAMPLE	3 h at 180°C	9 h at 180°C	24 h at 180°C	24 h at 230°C
Ti	220	462	619	785
Ti	56	433	565	820
Ti	4	4	6	77

Coating Campaign - TiZrHfV





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Conclusions

- Ageing Campaign

Ageing Campaign

- Some tested samples have shown problems during the activation

Ageing Campaign

- Some tested samples have shown problems during the activation

Coating Campaign

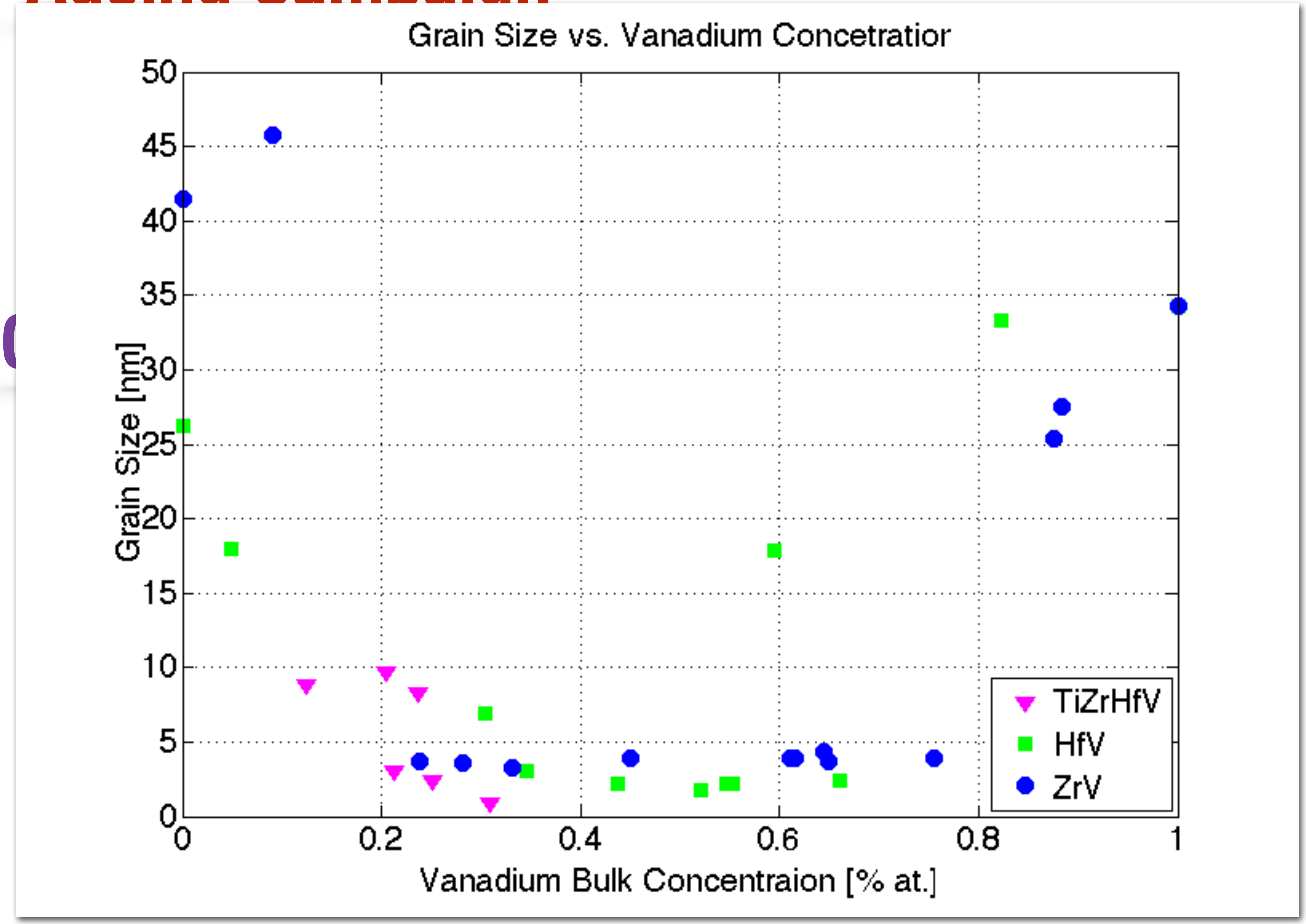
Ageing Campaign

- Some tested samples have shown problems during the activation

Coating Campaign

- The composition affects the activation temperature

Ageing Campaign



Ageing Campaign

- Some tested samples have shown problems during the activation

Coating Campaign

- The composition affects the activation temperature

Ageing Campaign

- Some tested samples have shown problems during the activation

Coating Campaign

- The composition affects the activation temperature
- The morphology affects the pumping properties

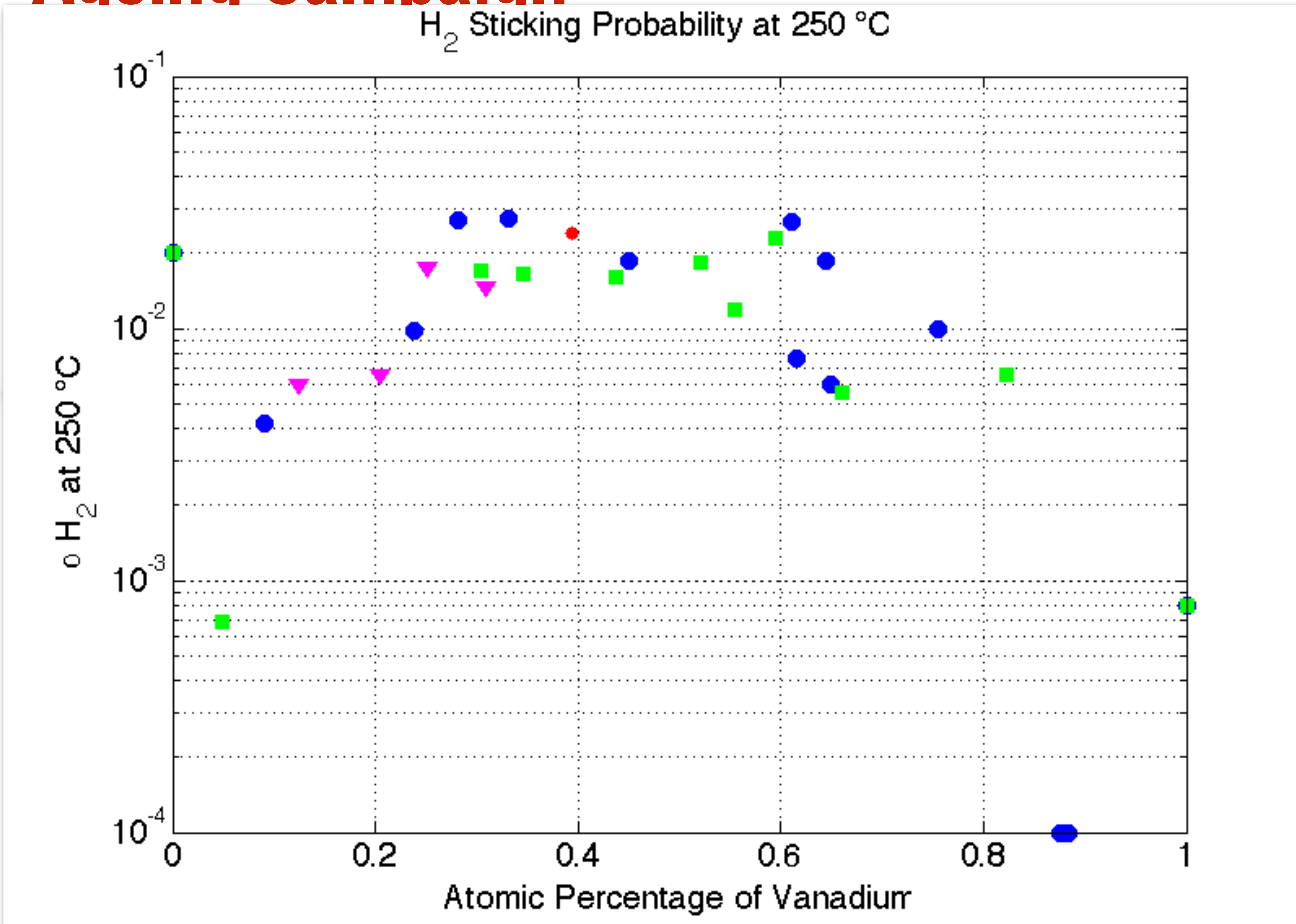
Ageing Campaign

- Some tested samples have shown problems during the activation

Coating Campaign

- The composition affects the activation temperature
- The morphology affects the pumping properties
- HfV alloys have a higher activation temperature than ZrV and TiZrHfV

Ageing Campaign



Thank you for your attention