

Non-Evaporable Getter Thin Film Coatings for Vacuum Application

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Supervisor: Paolo Chiggiato

- **NEG Materials Introduction**
- **Ageing Campaign**
- **Coating Campaign**
- **Conclusions**

Non-Evaporable Getter Materials Introduction

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Non-Evaporable Getter Materials Introduction



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



Non-Evaporable Getter Materials

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Sojourn Time

NEGs chemically react with the gases present in vacuum system



Non-Evaporable Getter Materials

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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

NEGs chemically react with the gases present in vacuum system

- free of contaminants
- free of the oxide layer



Non-Evaporable Getter Materials Introduction



Non-Evaporable Getter Materials Introduction



Active Surface



Non-Evaporable Getter Materials Introduction



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

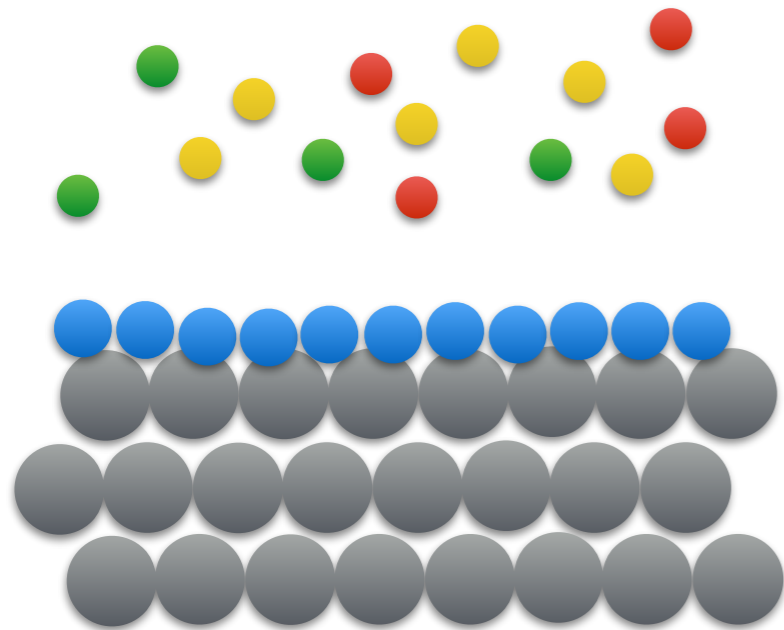
Non-Evaporable Getter Materials Introduction



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



Room Temperature

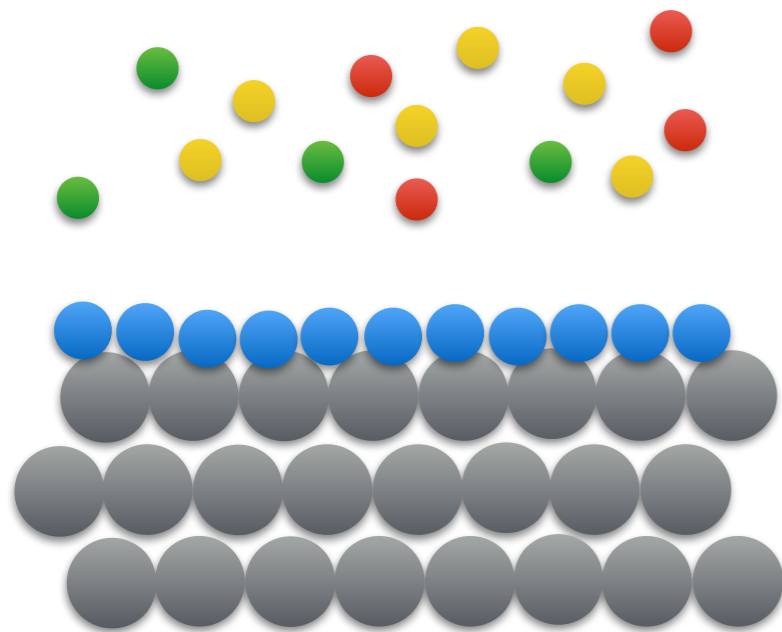
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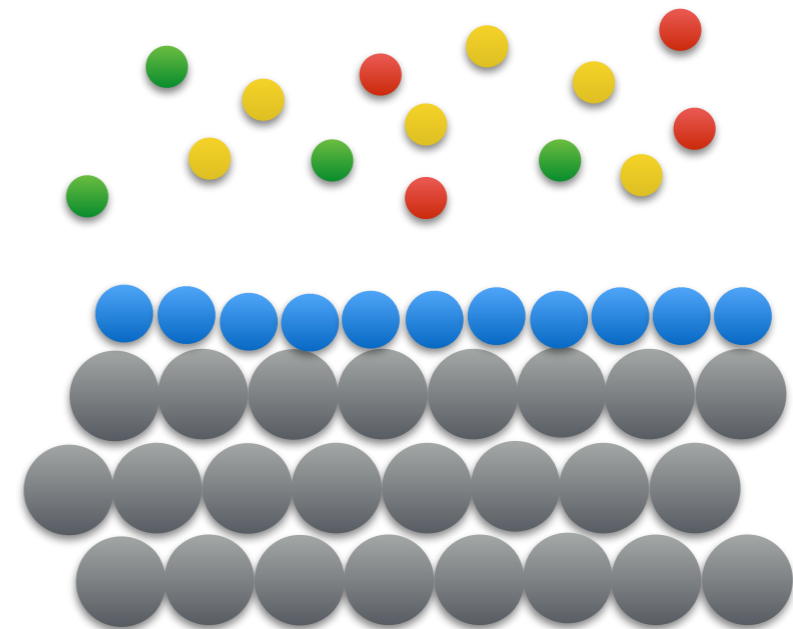
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Room Temperature



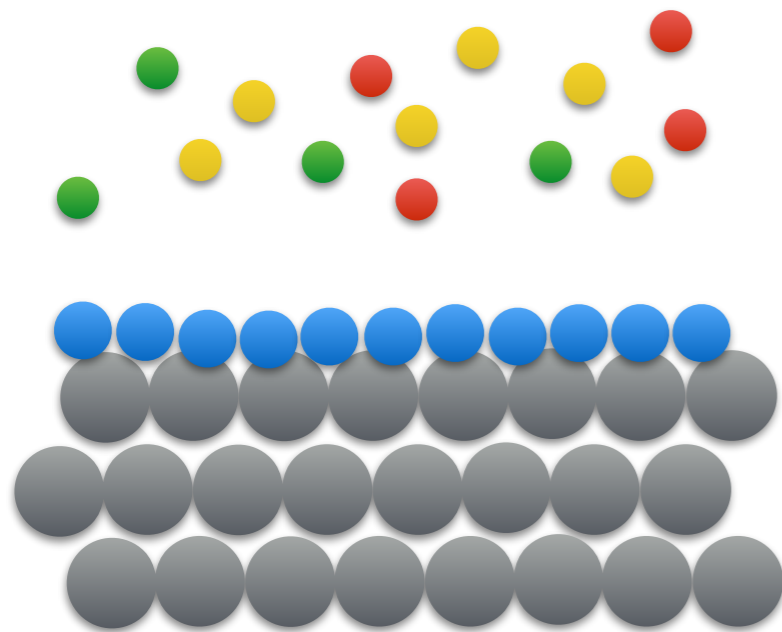
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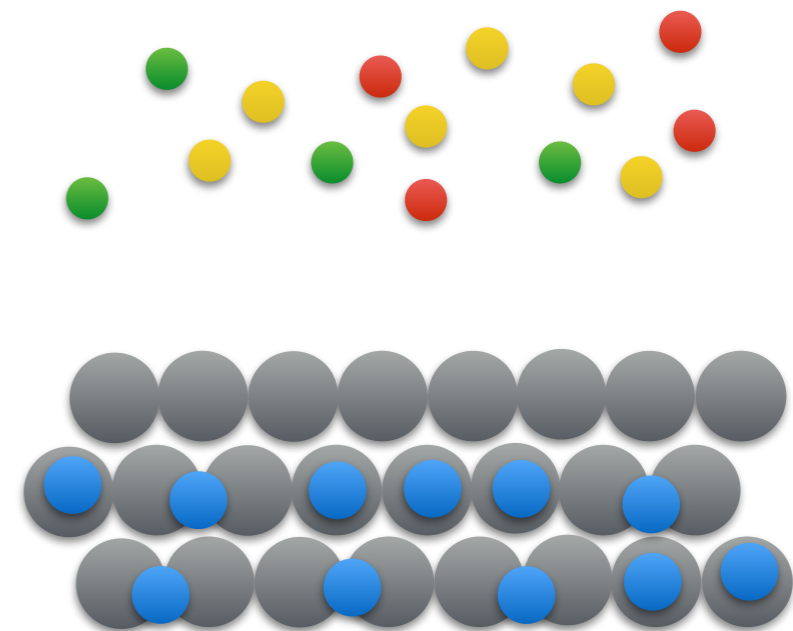
Active Surface



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



Room Temperature



Activation Temperature

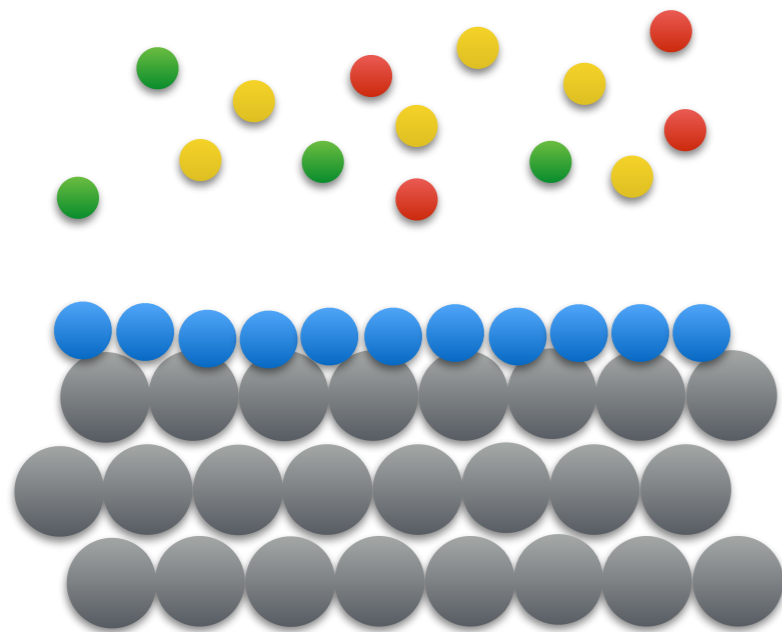
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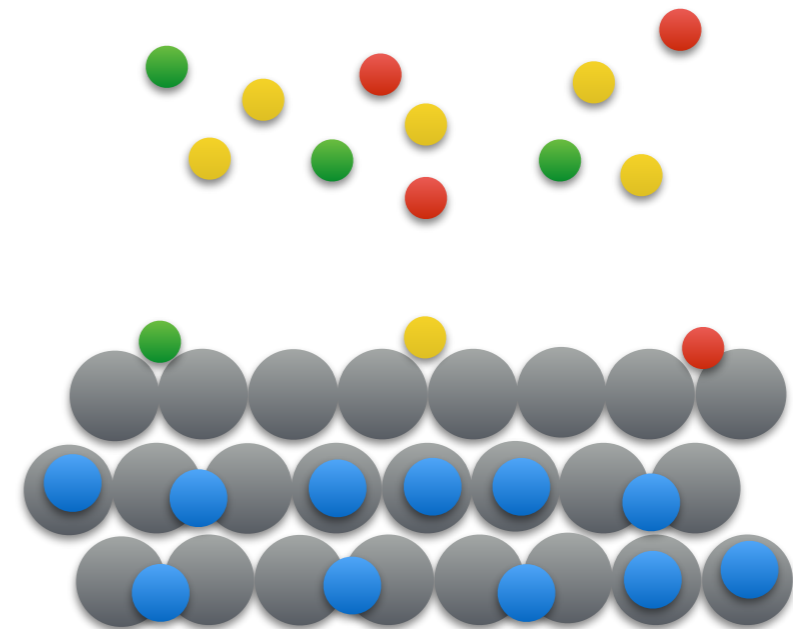
Active Surface



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



Room Temperature



Non-Evaporable Getter Materials Introduction



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**

Non-Evaporable Getter Materials Introduction



Suitable materials

1	2	13	14	15	16	17	18										
1 H Hydrogen		5 B Boron	6 C Carbon	7 N Nitrogen	8 O Oxygen	9 F Fluorine	10 Ne Neon										
3 Li Lithium	4 Be Beryllium																
11 Na Sodium	12 Mg Magnesium	3	4	5	6	7	8										
19 K Potassium	20 Ca Calcium	21 Sc Scandium	22 Ti Titanium	23 V Vanadium	24 Cr Chromium	25 Mn Manganese	26 Fe Iron	27 Co Cobalt	28 Ni Nickel	29 Cu Copper	30 Zn Zinc	31 Ga Gallium	32 Ge Germanium	33 As Arsenic	34 Se Selenium	35 Br Bromine	36 Kr Krypton
37 Rb Rubidium	38 Sr Strontium	39 Y Yttrium	40 Zr Zirconium	41 Nb Niobium	42 Mo Molybdenum	43 Tc Technetium	44 Ru Ruthenium	45 Rh Rhodium	46 Pd Palladium	47 Ag Silver	48 Cd Cadmium	49 In Indium	50 Sn Tin	51 Sb Antimony	52 Te Tellurium	53 I Iodine	54 Xe Xenon
55 Cs Cesium	56 Ba Barium	57-71 La-Lu Lanthanides	72 Hf Hafnium	73 Ta Tantalum	74 W Tungsten	75 Re Rhenium	76 Os Osmium	77 Ir Iridium	78 Pt Platinum	79 Au Gold	80 Hg Mercury	81 Tl Thallium	82 Pb Lead	83 Bi Bismuth	84 Po Polonium	85 At Astatine	86 Rn Radon
87 Fr Francium	88 Ra Radium	89-103 Ac-Lr Actinides	104 Rf Rutherfordium	105 Db Dubnium	106 Sg Seaborgium	107 Bh Bohrium	108 Hs Hassium	109 Mt Meitnerium	110 Ds Darmstadtium	111 Rg Roentgenium	112 Uub Ununbium	113 Uut Ununtrium	114 Uuq Ununquadium	115 Uup Ununpentium	116 Uuh Ununhexium	117 Uus Ununseptium	118 Uuo Ununoctium
Lanthanides		57 La Lanthanum	58 Ce Cerium	59 Pr Praseodymium	60 Nd Neodymium	61 Pm Promethium	62 Sm Samarium	63 Eu Europium	64 Gd Gadolinium	65 Tb Terbium	66 Dy Dysprosium	67 Ho Holmium	68 Er Erbium	69 Tm Thulium	70 Yb Ytterbium	71 Lu Lutetium	
Actinides		89 Ac Actinium	90 Th Thorium	91 Pa Protactinium	92 U Uranium	93 Np Neptunium	94 Pu Plutonium	95 Am Americium	96 Cm Curium	97 Bk Berkelium	98 Cf Californium	99 Es Einsteinium	100 Fm Fermium	101 Md Mendelevium	102 No Nobelium	103 Lr Lawrencium	

Non-Evaporable Getter Materials Introduction



Suitable materials

IV group elements

1	2											13	14	15	16	17	18												
1 H												5 B	6 C	7 N	8 O	9 F	10 Ne												
2 3 Li	4 Be											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar												
3 11 Na	12 Mg											19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
4 19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr												
5 37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe												
6 55 Cs	56 Ba	57-71 La-Lu	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn												
7 87 Fr	88 Ra	89-103 Ac-Lr	104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Uub	113 Uut	114 Uuq	115 Uup	116 Uuh	117 Uus	118 Uuo												
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Actinides		89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr													

Non-Evaporable Getter Materials Introduction



Suitable materials

IV group elements

- high solubility limit: 30 at.%
- low diffusivity

1	2	13	14	15	16	17	18									
1 H							2 He									
3 Li	4 Be						10 Ne									
11 Na	12 Mg	3	4				18 Ar									
19 K	20 Ca	Sc	Ti				36 Kr									
37 Rb	38 Sr	Y	Zr	Nb	Mo		54 Xe									
55 Cs	56 Ba	La-L	Hf	Ta	W	Re	86 Rn									
87 Fr	88 Ra	Ac-L	Rf	Db	Sg	Bh	118 Uuo									
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Non-Evaporable Getter Materials Introduction



Suitable materials

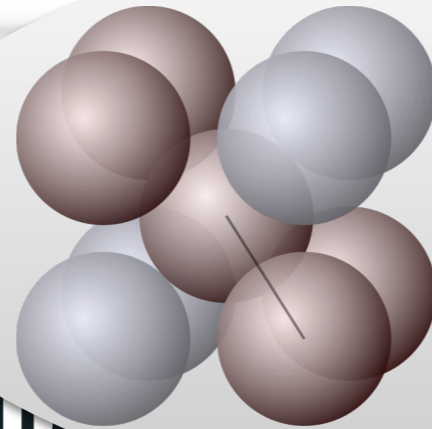
V group elements

1	2											13	14	15	16	17	18	
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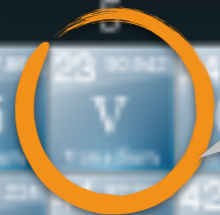


Suitable



- low solubility limit
- high diffusivity

V group



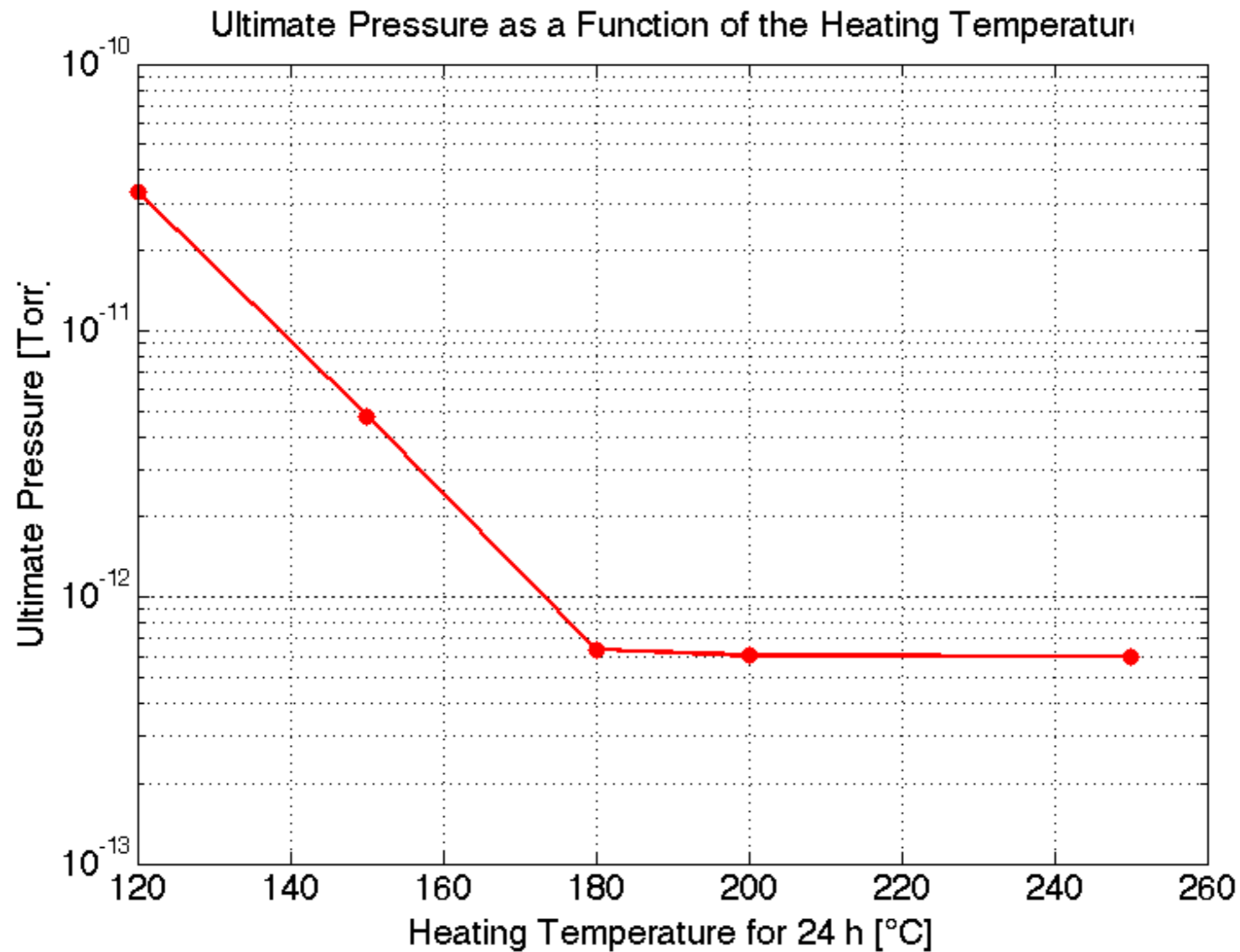
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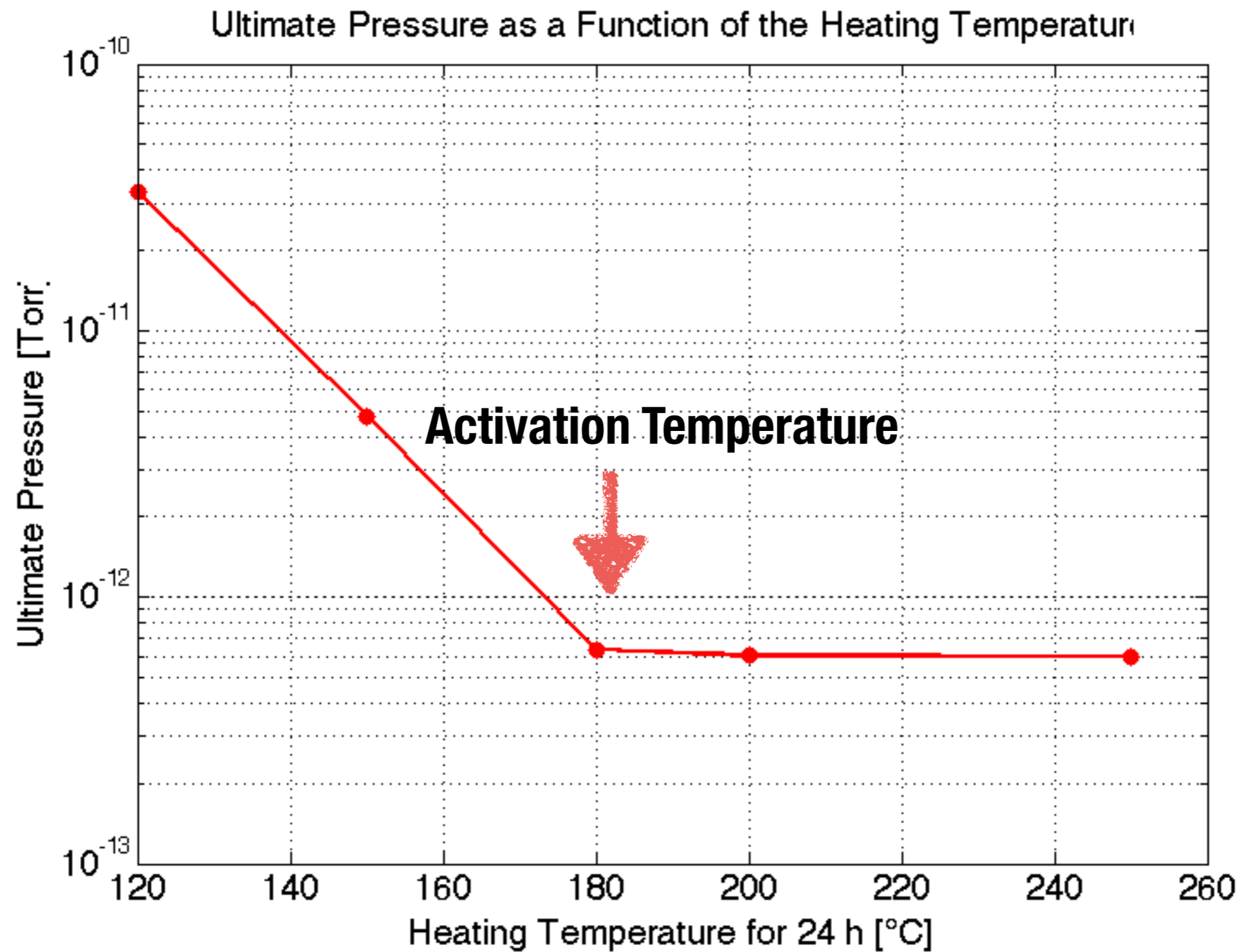


Ti₃₀Zr₃₀V₄₀ is fully activated after **24 h** of heating at **180 °C**

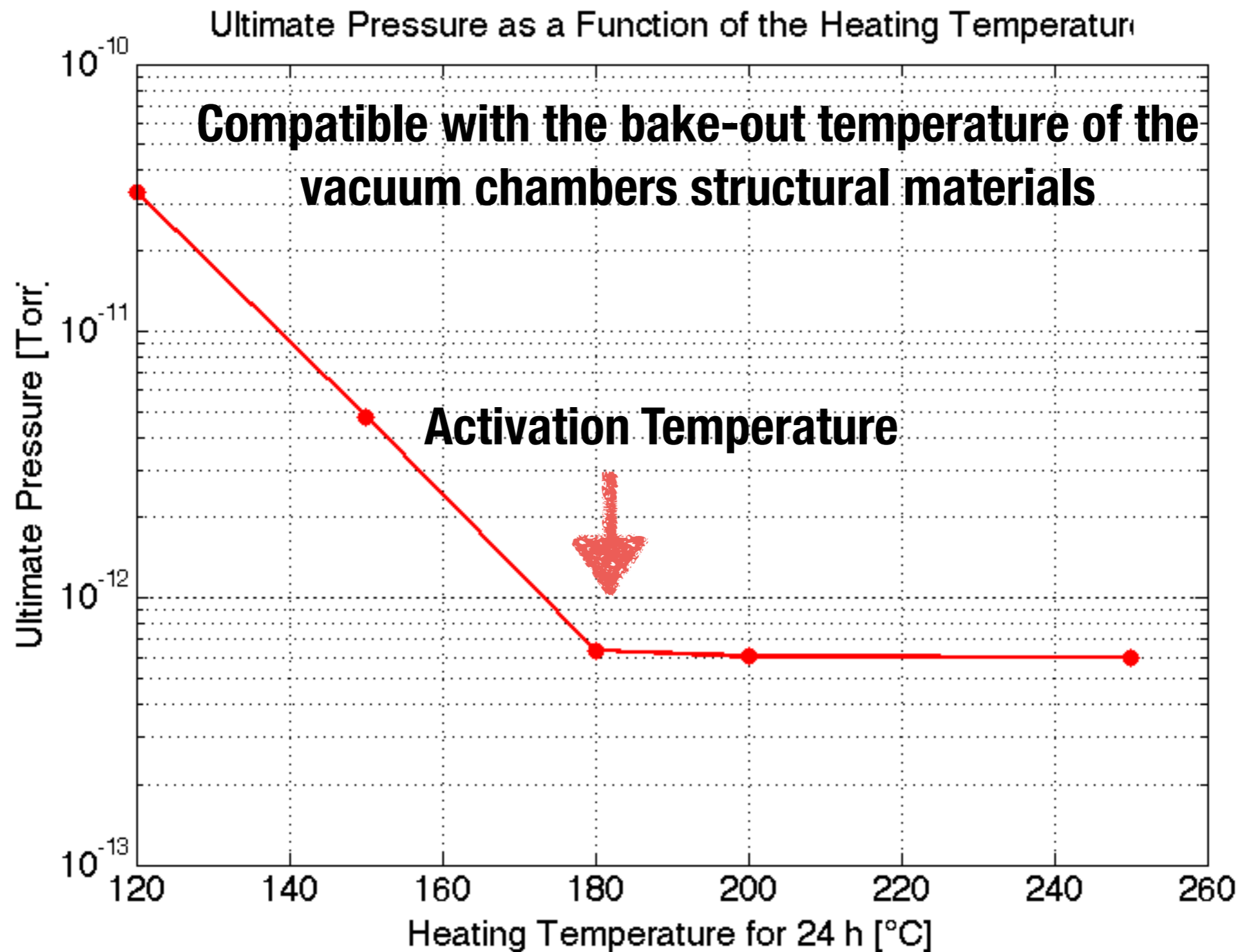
Non-Evaporable Getter Materials Introduction



Non-Evaporable Getter Materials Introduction



Non-Evaporable Getter Materials Introduction



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

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

Ageing Campaign

- **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?**

- **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

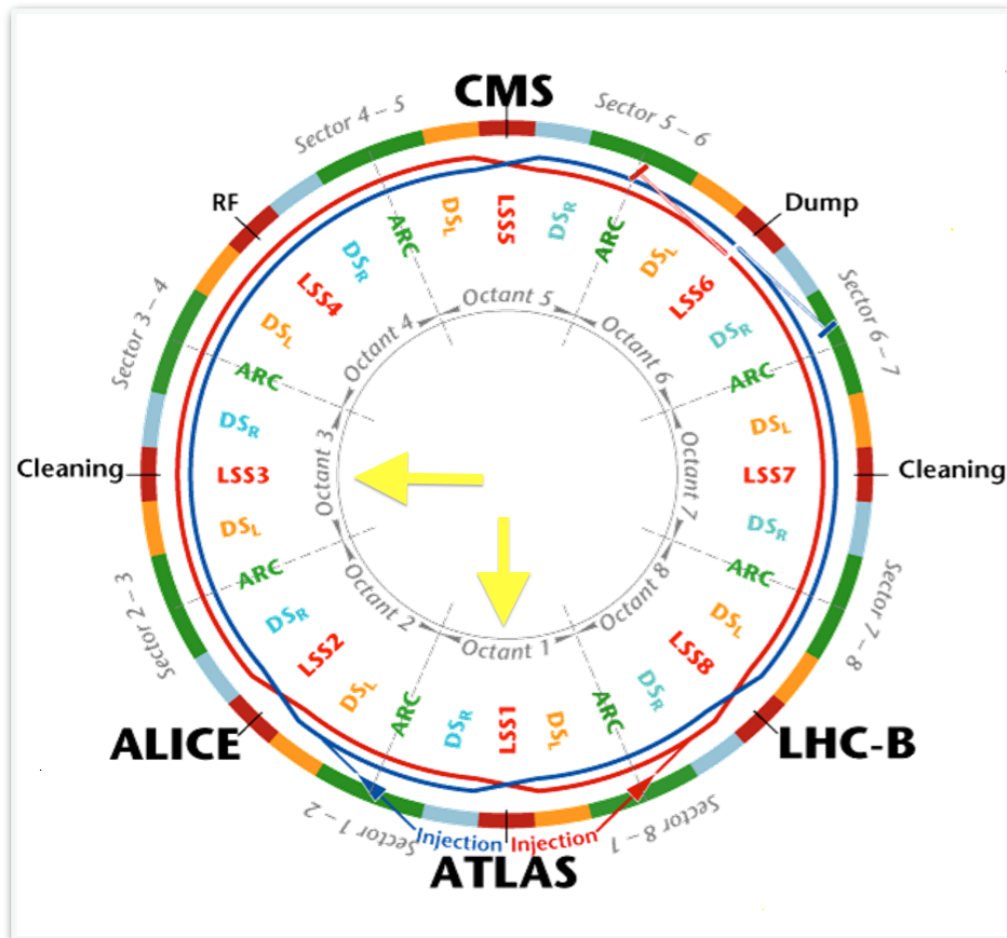
Ageing Campaign

Exposure Points

Ageing Campaign



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



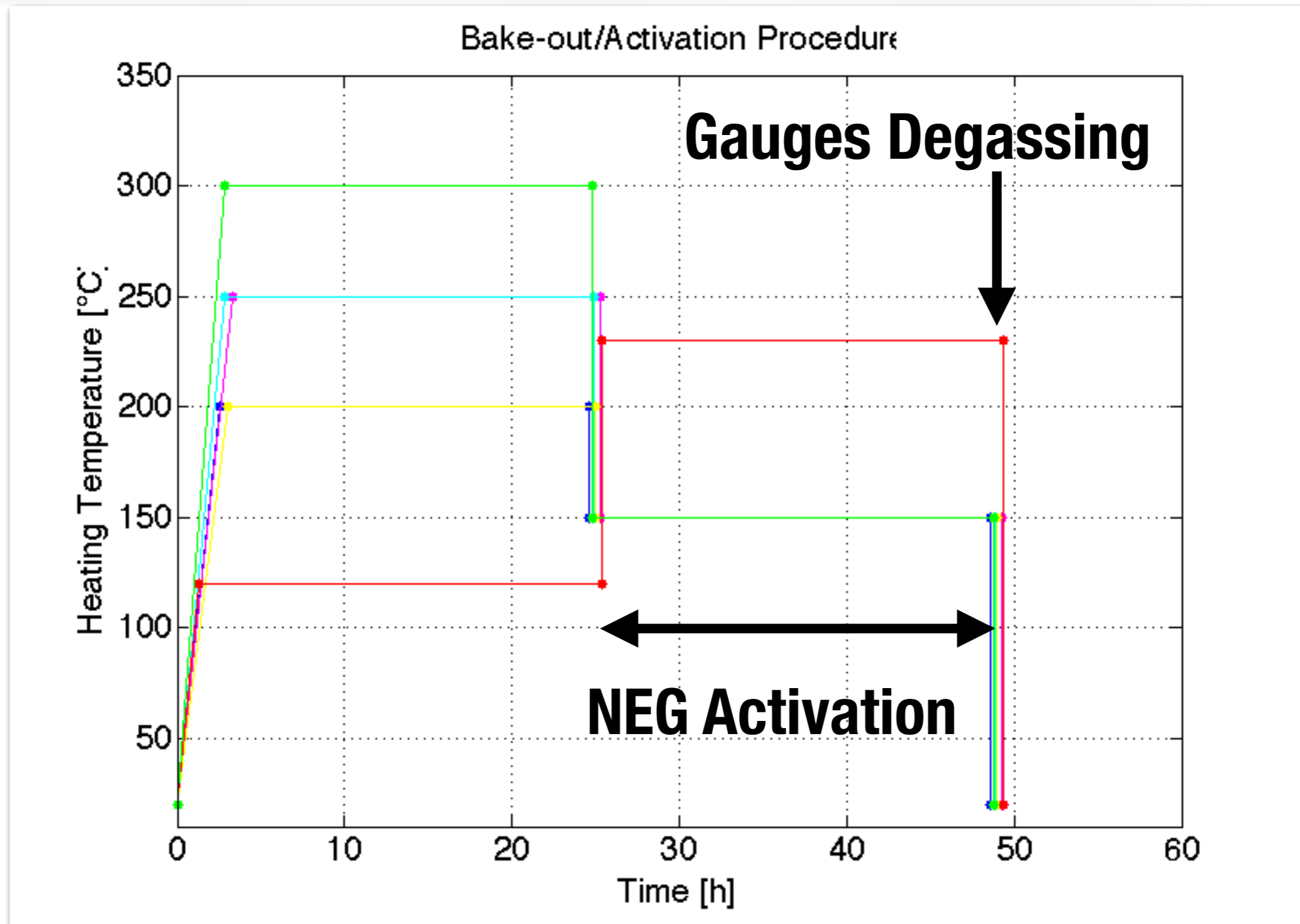


Bake-out/Activation Procedure

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

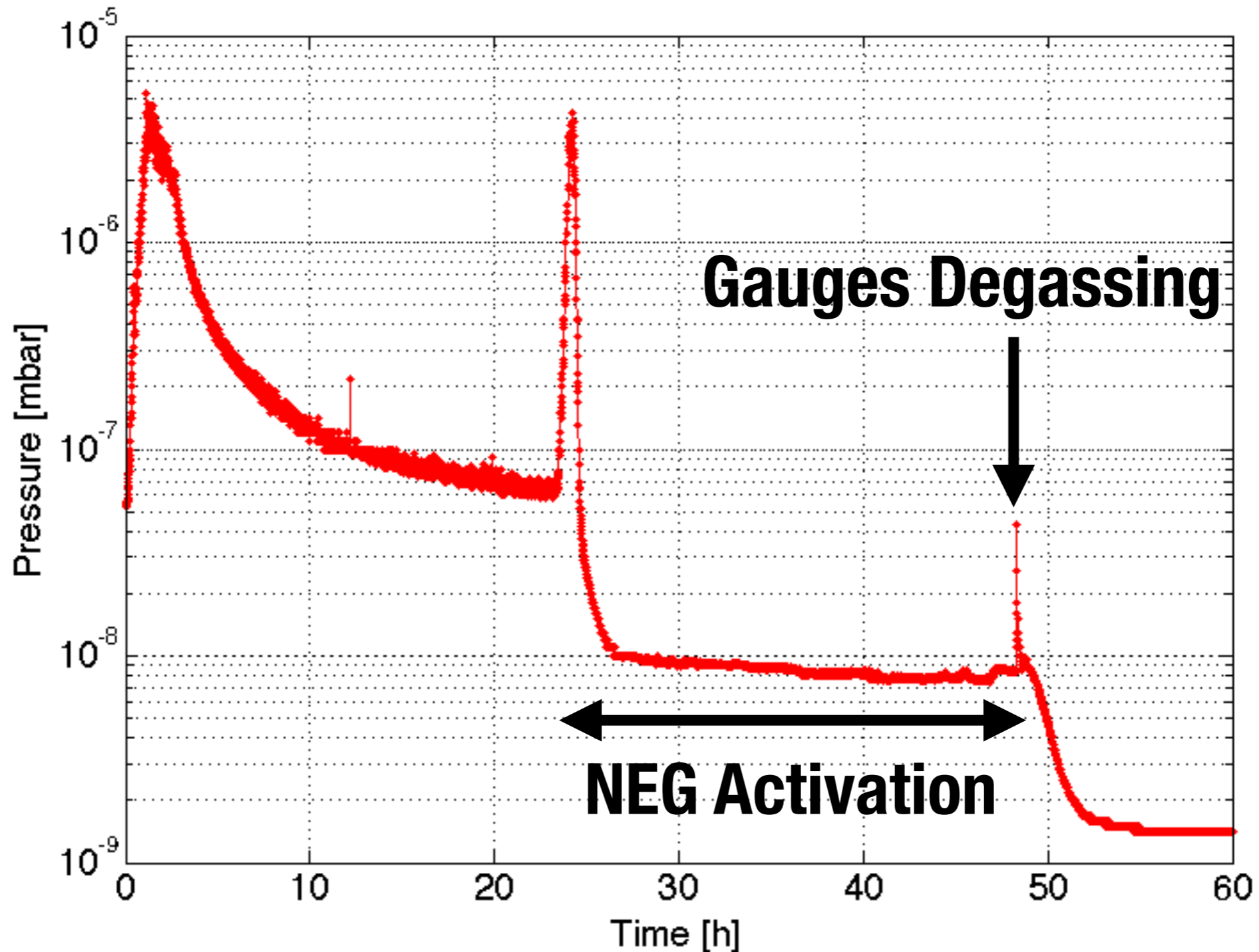


Bake-out/Activation Procedure



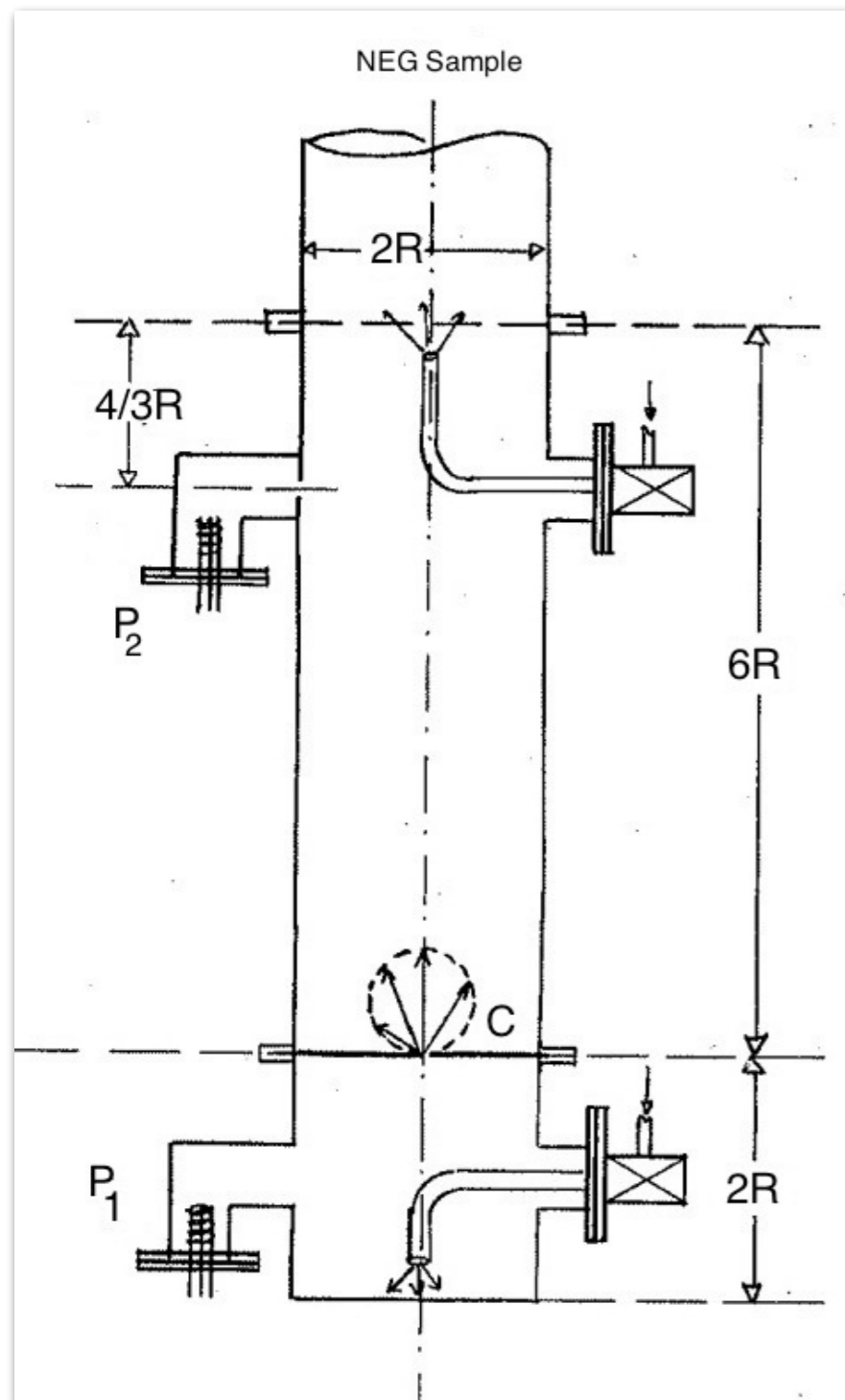


Bake-out/Activation Procedure





Fischer-Mommensen Dome



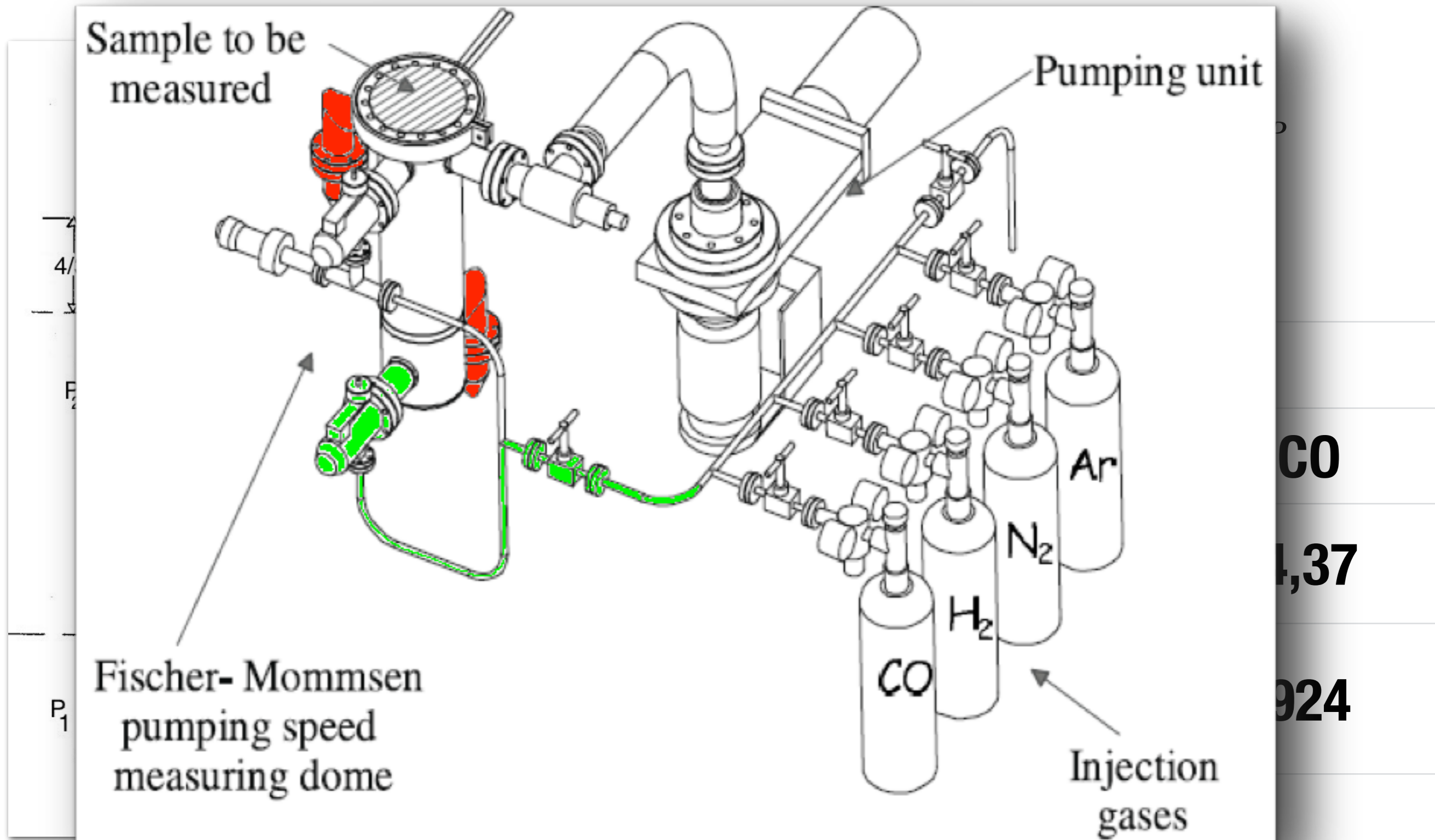
$$S_{eff}^{NEG} = C \left(\frac{\Delta P_1}{\Delta P_2} - 1 \right) - S_{eff}^{TMP}$$

System Parameters

	H	CO
S	8	4,37
S	3456	924



Fischer-Mommensen Dome



Experimental Results

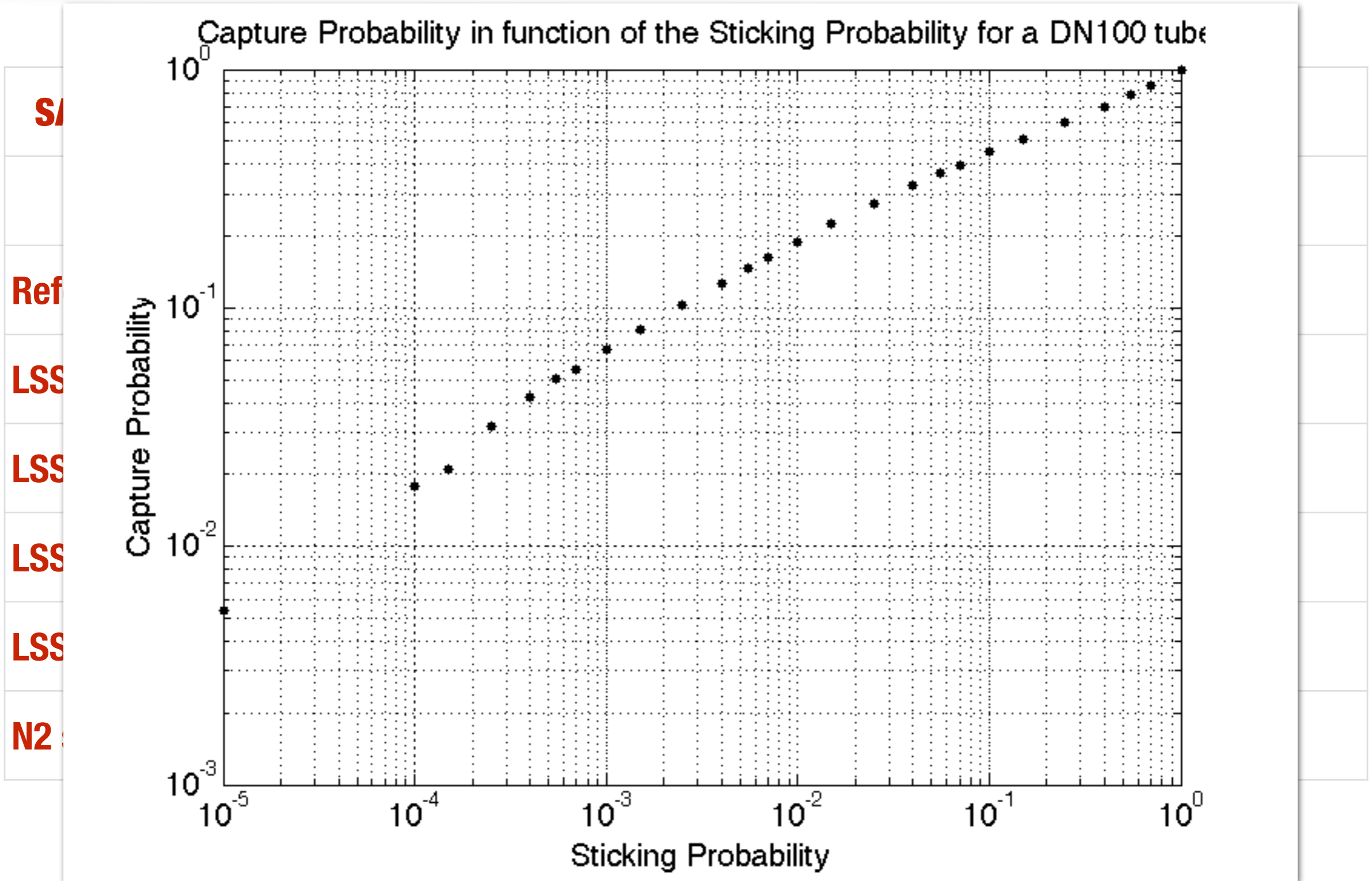
Ageing Campaign



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

Experimental Results

Ageing Campaign



SAMPLE	H2 Pumping Speed [l/s]			Sticking Probability		
	I Act	II Act	Vented	I Act	II Act	Vented
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Ageing Campaign



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LSS1_1	615	-	669	1 10	-	1 10
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Experimental Results

Ageing Campaign



SAMPLE	I Activation		II Activation		Vented	
	V1 [Torr]	V2 [Torr]	V1 [Torr]	V2 [Torr]	V1 [Torr]	V2 [Torr]
R Reference	7,7 10 ⁻¹¹	1,52 10 ⁻¹¹	-	-	-	-
L LSS3_1	-	-	5 10 ⁻¹¹	1,3 10 ⁻¹⁰	2,65 10 ⁻¹⁰	9,3 10 ⁻¹¹
L LSS3_2	5 10 ⁻¹¹	1,1 10 ⁻¹⁰	-	-	-	-
L LSS1_1	1,80 10 ⁻⁹	3,5 10 ⁻⁹	-	-	1,61 10 ⁻⁹	3,6 10 ⁻⁹
L LSS1_2	NOT TESTED					
N N2 sealed	NOT LEAK TIGHT					

Experimental Results

Ageing Campaign



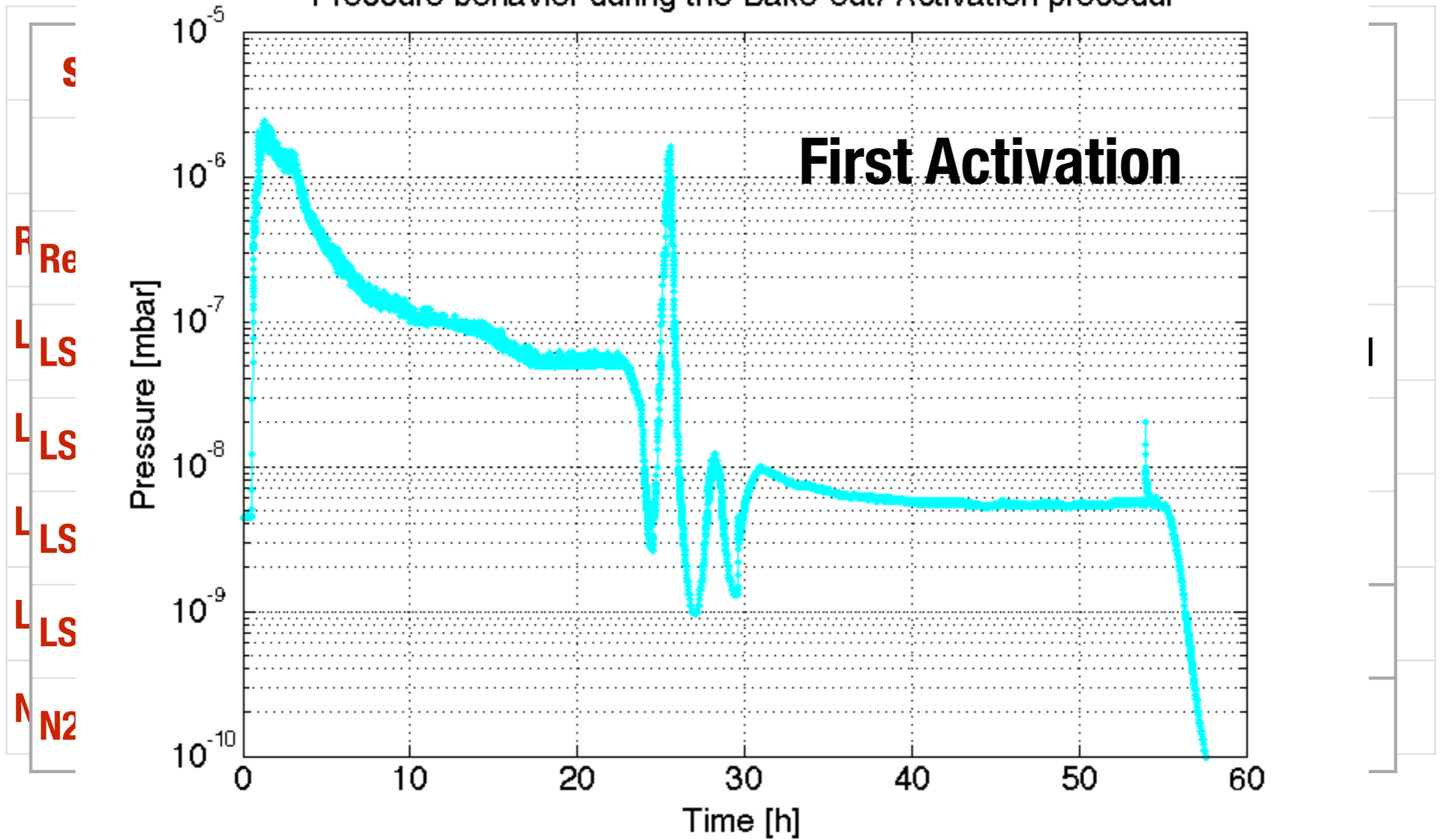
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	V1 [Torr]	V2 [Torr]	V1 [Torr]	V2 [Torr]	V1 [Torr]	V2 [Torr]
R Reference	7,7 10 ⁻¹¹	1,52 10 ⁻¹¹	-	-	-	-
L LSS3_1	-	-	5 10 ⁻¹¹	1,3 10 ⁻¹⁰	2,65 10 ⁻¹⁰	9,3 10 ⁻¹¹
L LSS3_2	5 10 ⁻¹¹	1,1 10 ⁻¹⁰	-	-	-	-
L LSS1_1	1,80 10 ⁻⁹	3,5 10 ⁻⁹	-	-	1,61 10 ⁻⁹	3,6 10 ⁻⁹
L LSS1_2	NOT TESTED					
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Experimental Results

Ageing Campaign



Pressure behavior during the Bake-out/ Activation procedur



Experimental Results

Ageing Campaign



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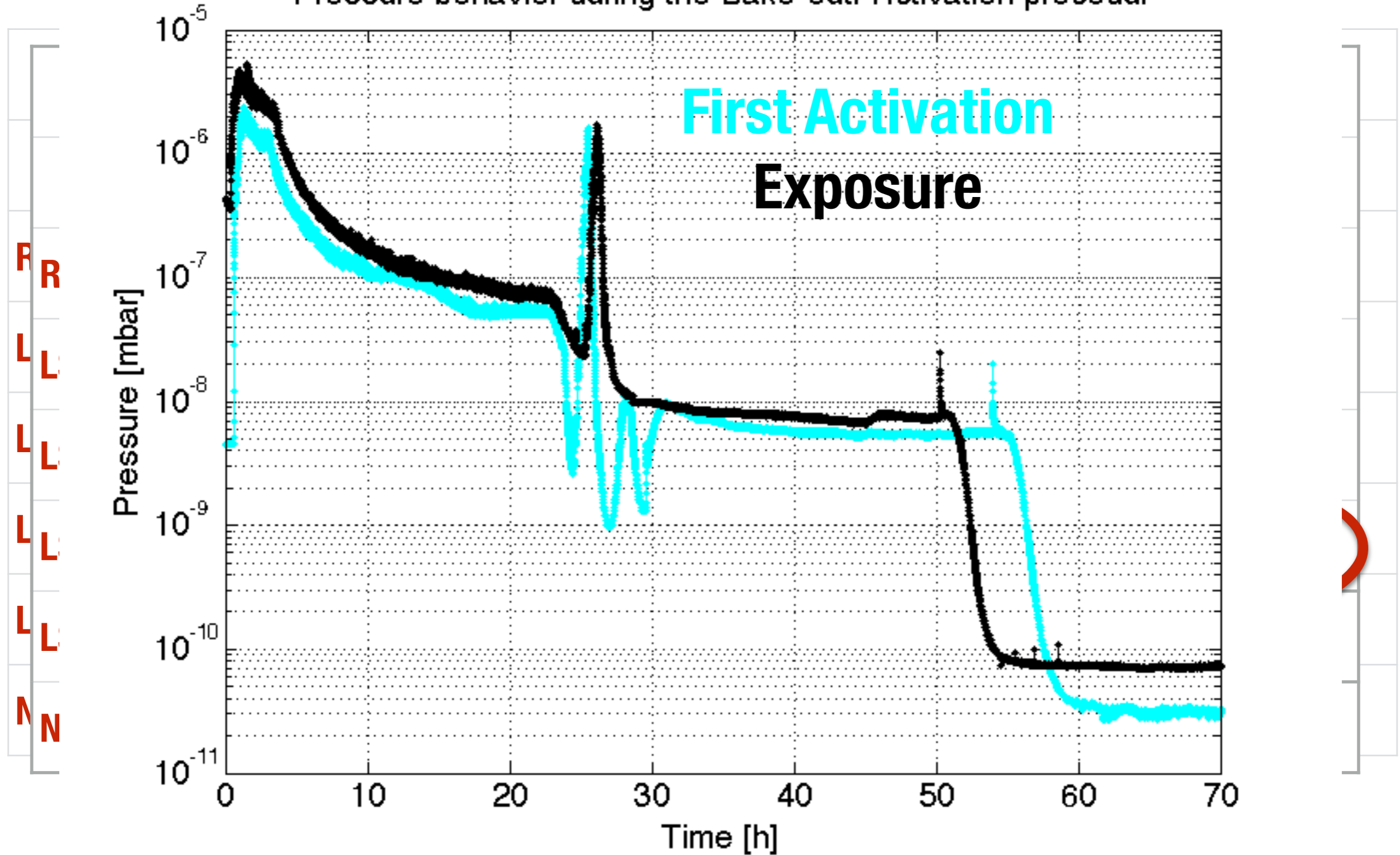
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L LSS3_1	-	-	5 10 ⁻¹¹	1,3 10 ⁻¹⁰	2,65 10 ⁻¹⁰	9,3 10 ⁻¹¹
L LSS3_2	5 10 ⁻¹¹	1,1 10 ⁻¹⁰	-	-	-	-
L LSS1_1	1,80 10 ⁻⁹	3,5 10 ⁻⁹	-	-	1,61 10 ⁻⁹	3,6 10 ⁻⁹
L LSS1_2	NOT TESTED					
N N2 sealed	NOT LEAK TIGHT					

Experimental Results

Ageing Campaign



Pressure behavior during the Bake-out/ Activation procedur



Experimental Results

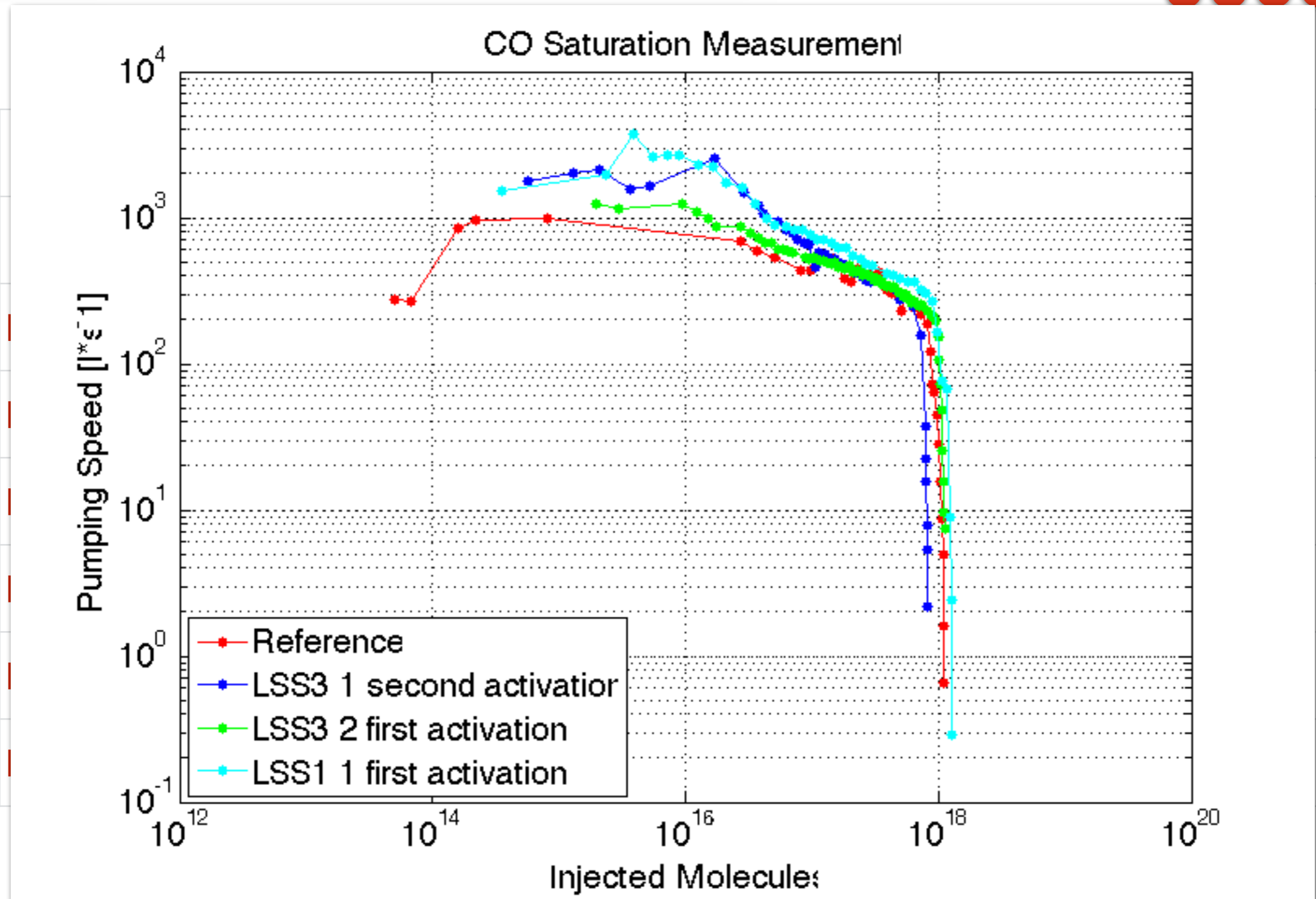
Ageing Campaign



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

Ageing Campaign



Coating Campaign



Compositions tested

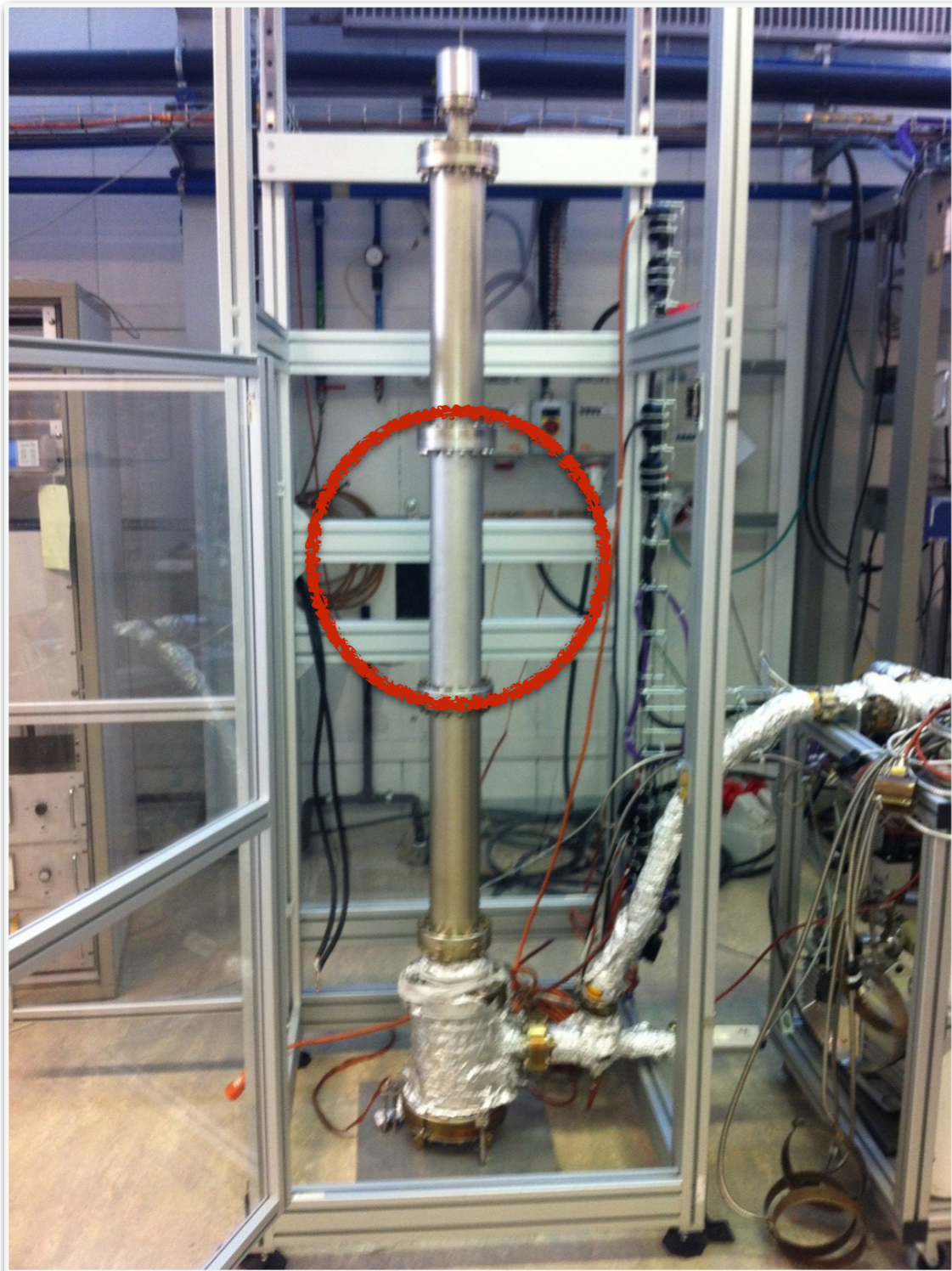
- ZrV
- HfV
- TiZrHfV

Experimental Set-Up

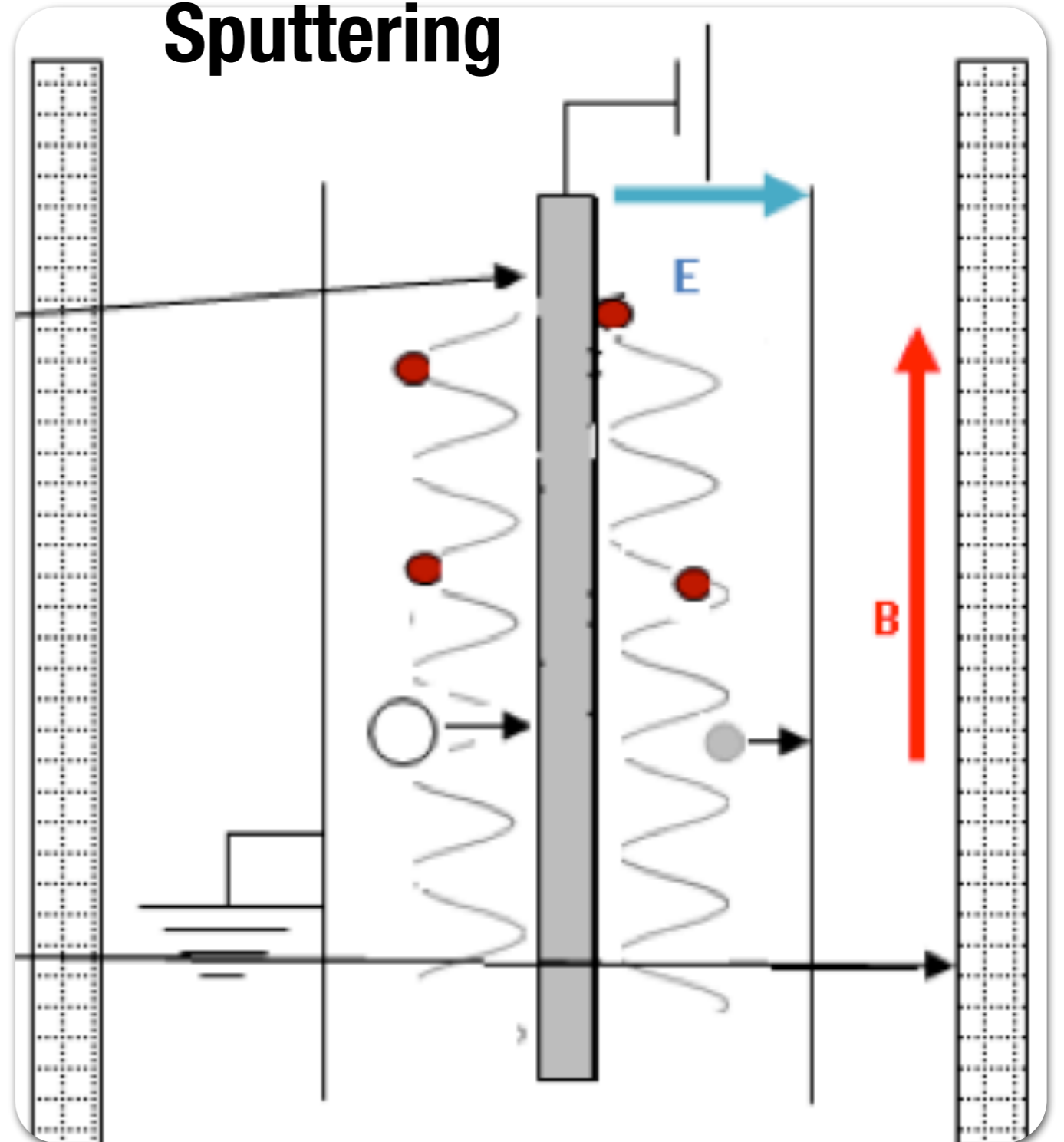
Coating Campaign



Coating Tower



Magnetron Sputtering

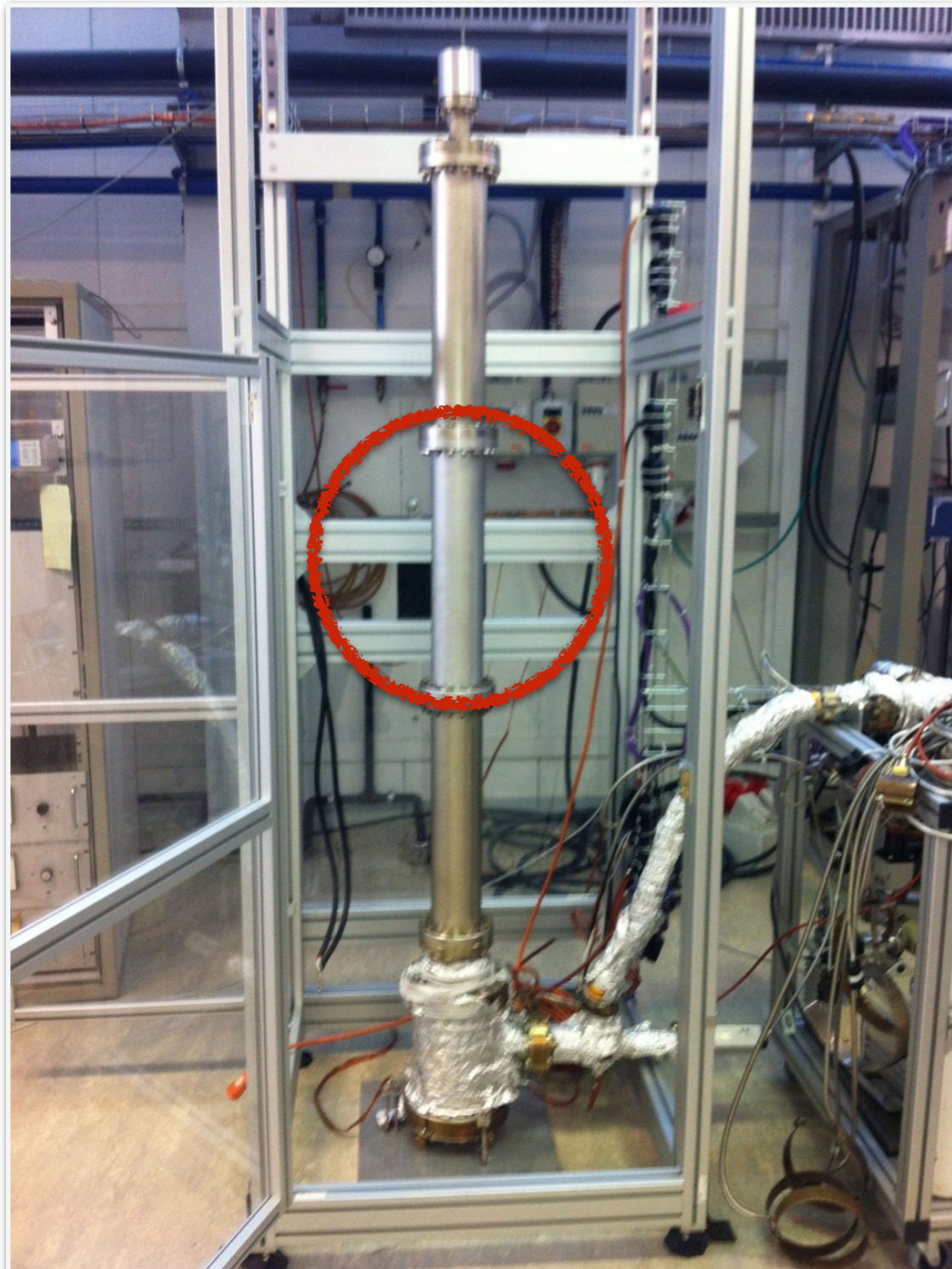


Experimental Set-Up

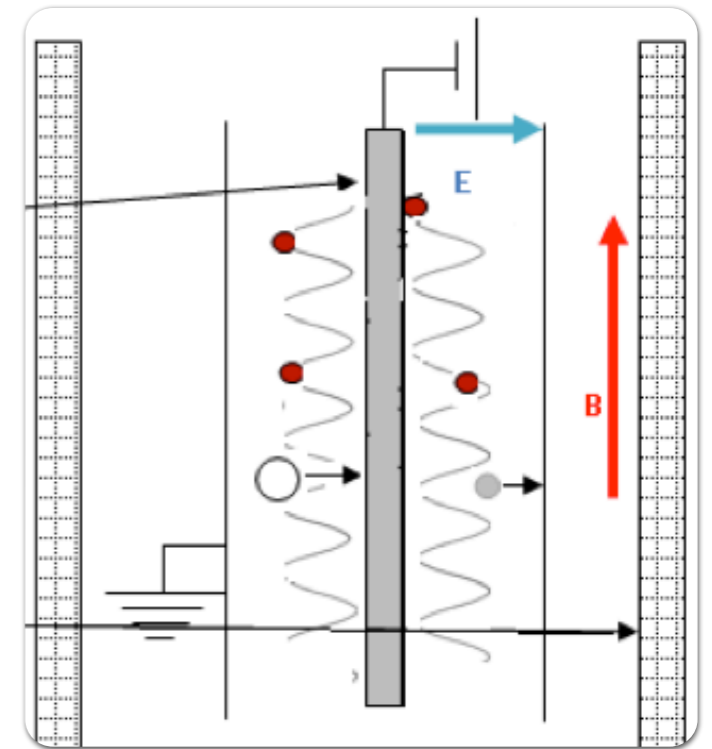
Coating Campaign



Coating Tower



Magnetron Sputtering



Coating Parameters

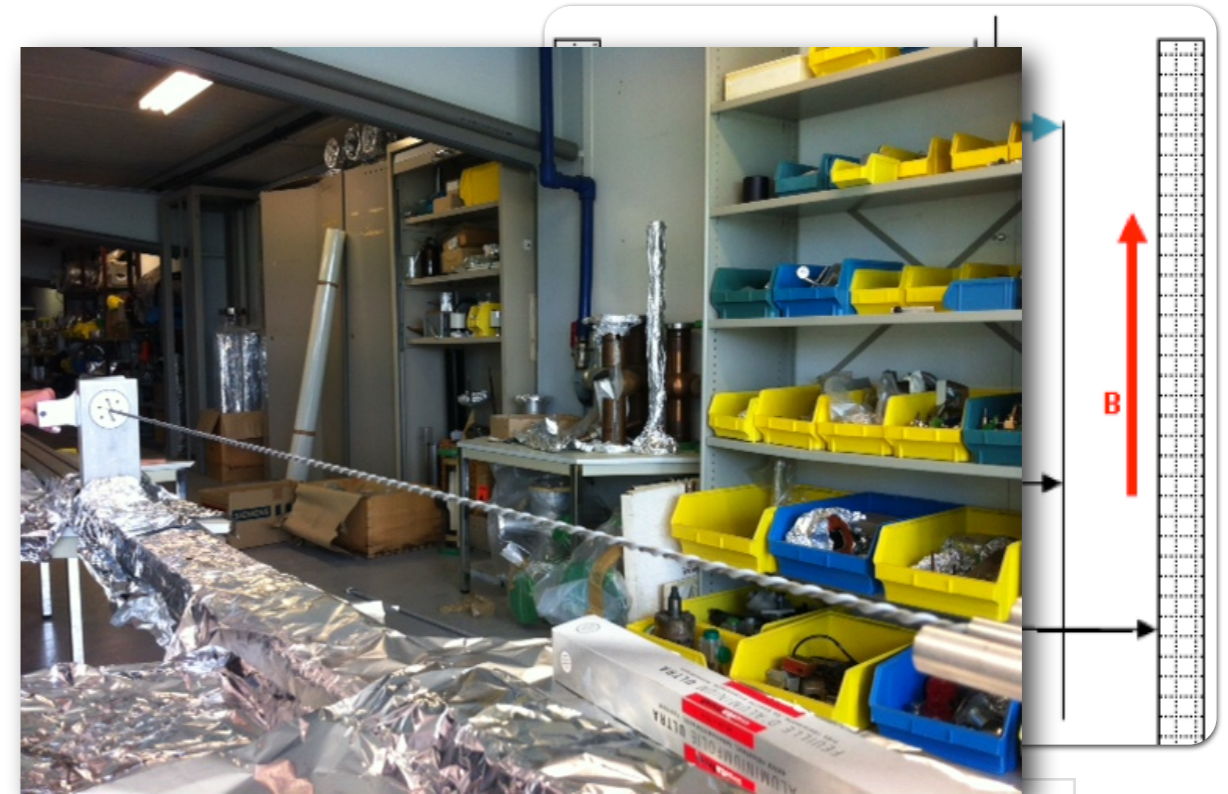
Discharge Gas	kr
Gas Pressure	4,5 10 ⁻³ Torr
Solenoid Current	~ 100 A
Solenoid Potential	~ 40 V
Cathode Current	- 600 V
Cathode Potential	0,1 kW (DC)
Cathode Power	0,2 A
Coating Tower Temperature	100 °C

Experimental Set-Up

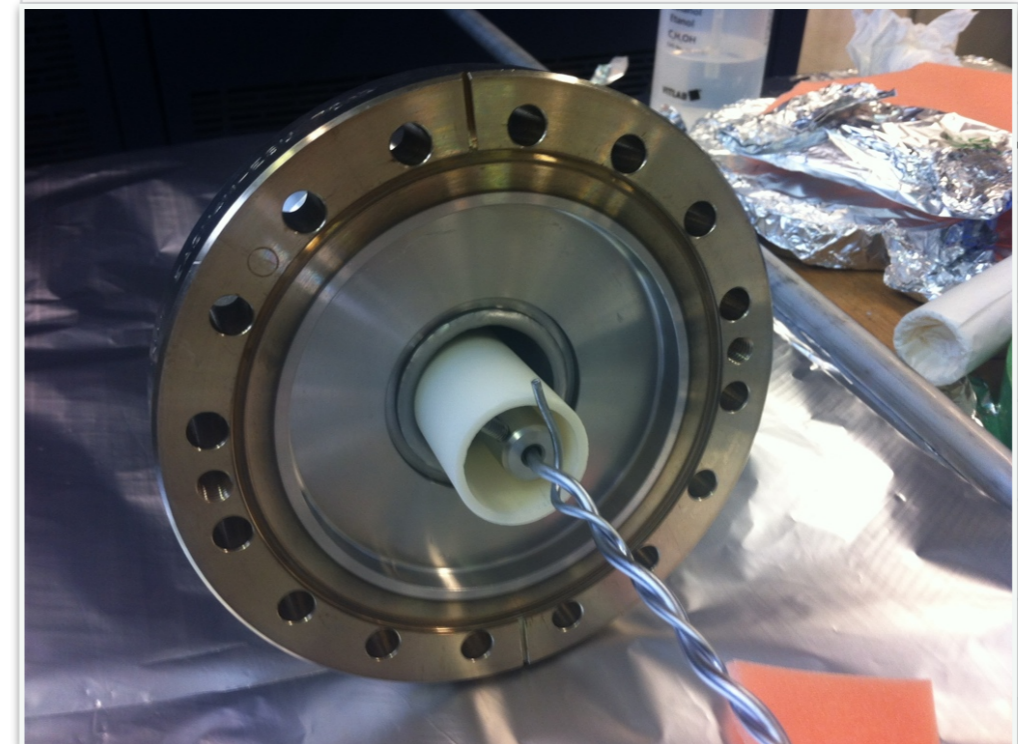
Coating Campaign



Coating Tower



Coating Parameters



Coating tower temperature

100 °C

Characterization Measurements



Macroscopic Measurement

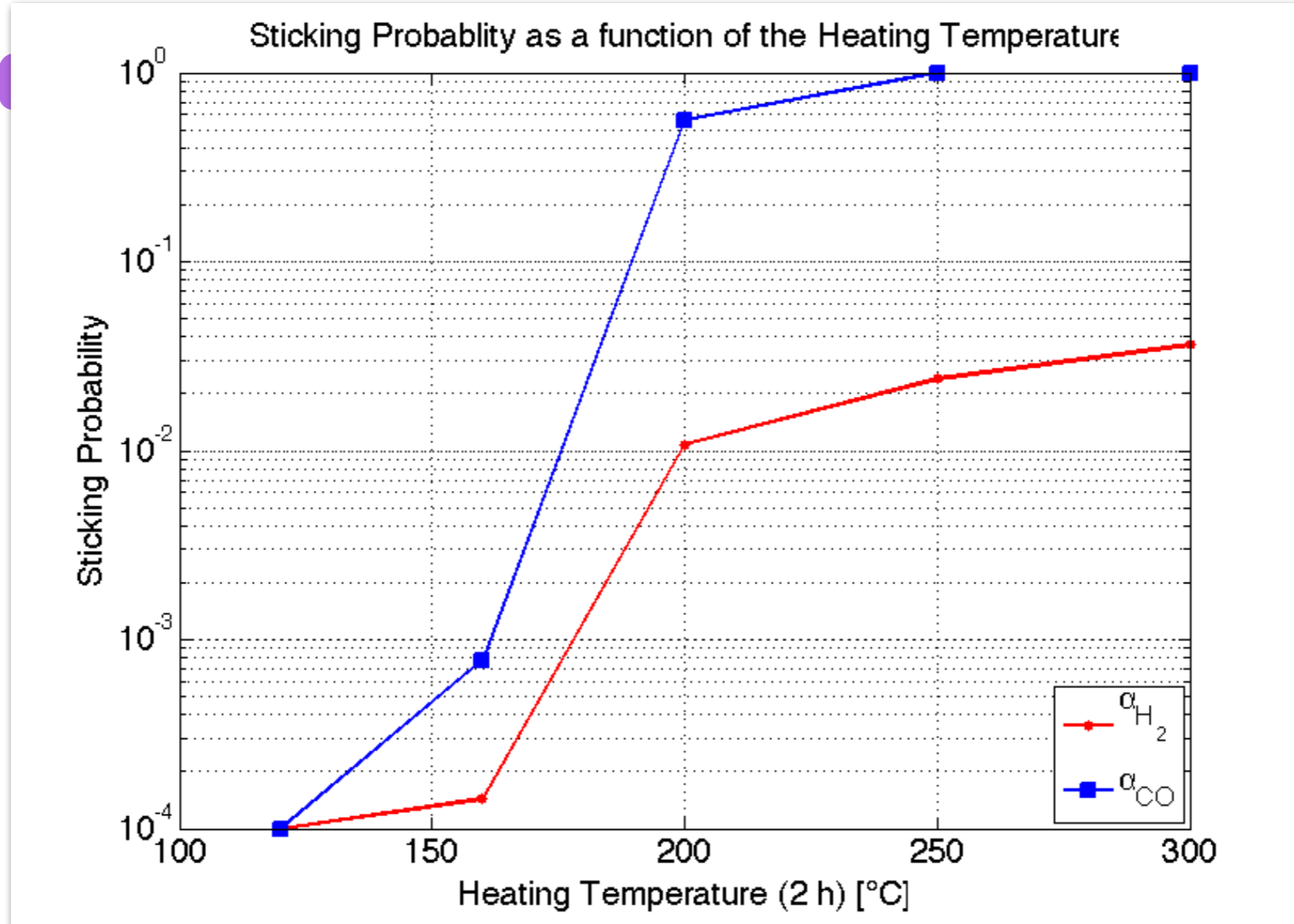
- Sticking Probability for H₂ and CO

Characterization Measurements



Macroscopic Measurement

● Sticking



Characterization Measurements



Macroscopic Measurement

- Sticking Probability for H₂ and CO

Characterization Measurements



Macroscopic Measurement

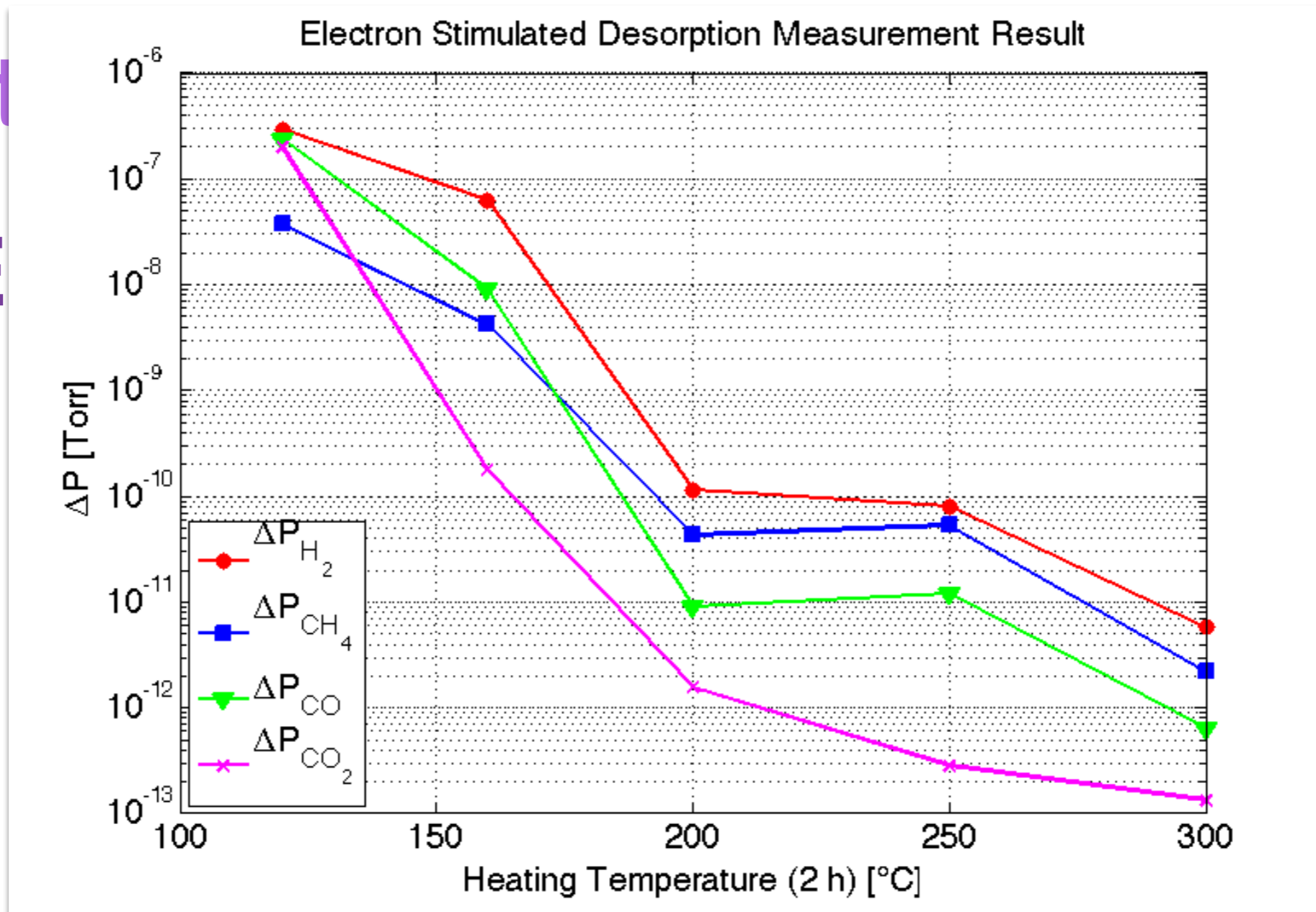
- Sticking Probability for H₂ and CO
- Electron Stimulated Desorption - ESD

Characterization Measurements



Macroscopic Measurement

- St
- E



D

Characterization Measurements



Macroscopic Measurement

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

Characterization Measurements



Macroscopic Measurement

- **Sticking Probability for H₂ and CO**
- **Electron Stimulated Desorption - ESD**
- **Pumping Speed Measurement**

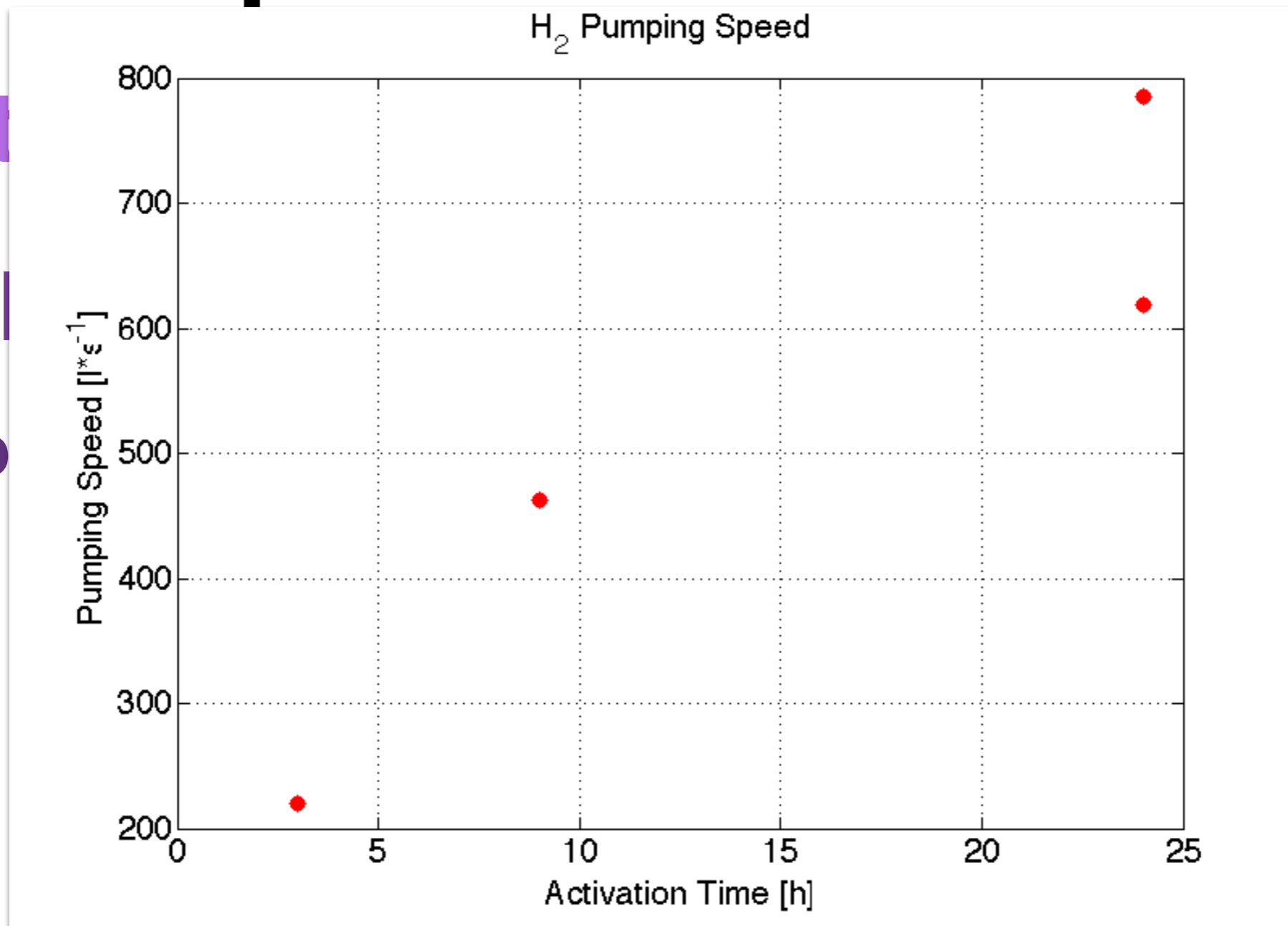
Characterization Measurements

Coating Campaign



Macroscopic Measurement

- St
- El
- P



D

Characterization Measurements



Macroscopic Measurement

- **Sticking Probability for H₂ and CO**
- **Electron Stimulated Desorption - ESD**
- **Pumping Speed Measurement**

Characterization Measurements

Microscopic Measurement

Coating Campaign

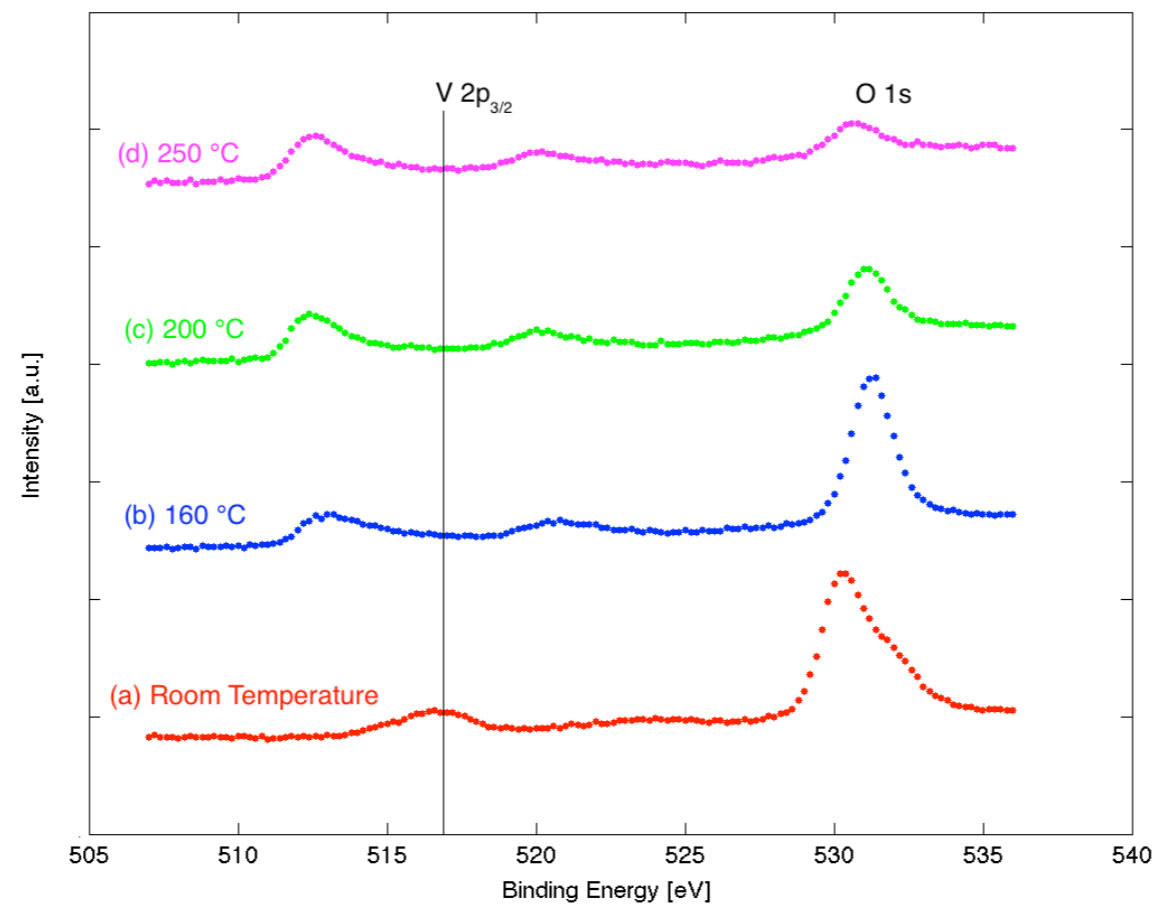


Characterization Measurements



Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS



Characterization Measurements

Coating Campaign



Microscopic Measurement

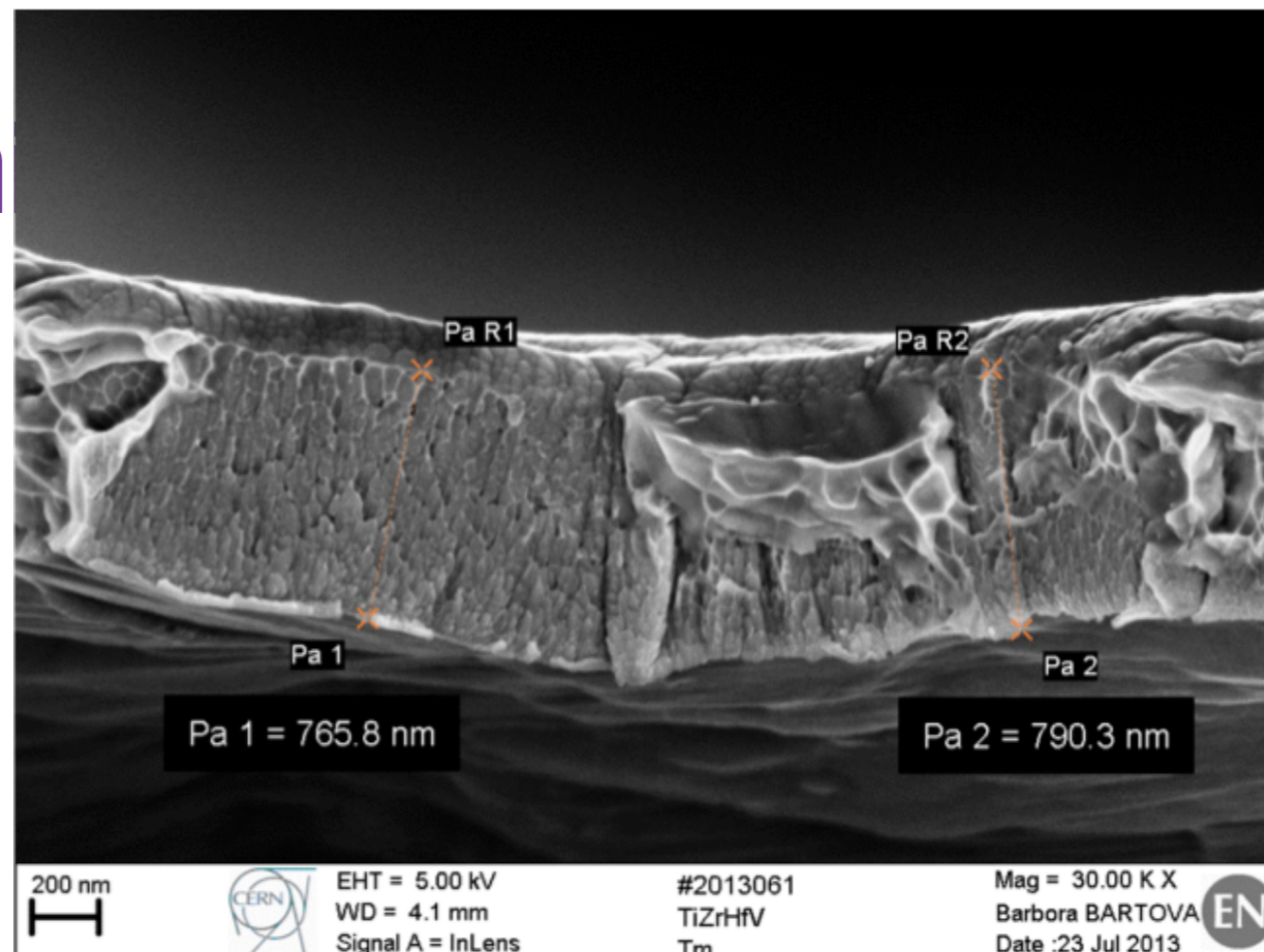
- X-Rays Photoelectron Spectroscopy - XPS
- Scanning Electron Microscopy - SEM

Characterization Measurements



Microscopic Measurement

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



Characterization Measurements

Coating Campaign



Microscopic Measurement

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

Characterization Measurements

Coating Campaign



Microscopic Measurement

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

Characterization Measurements

Coating Campaign

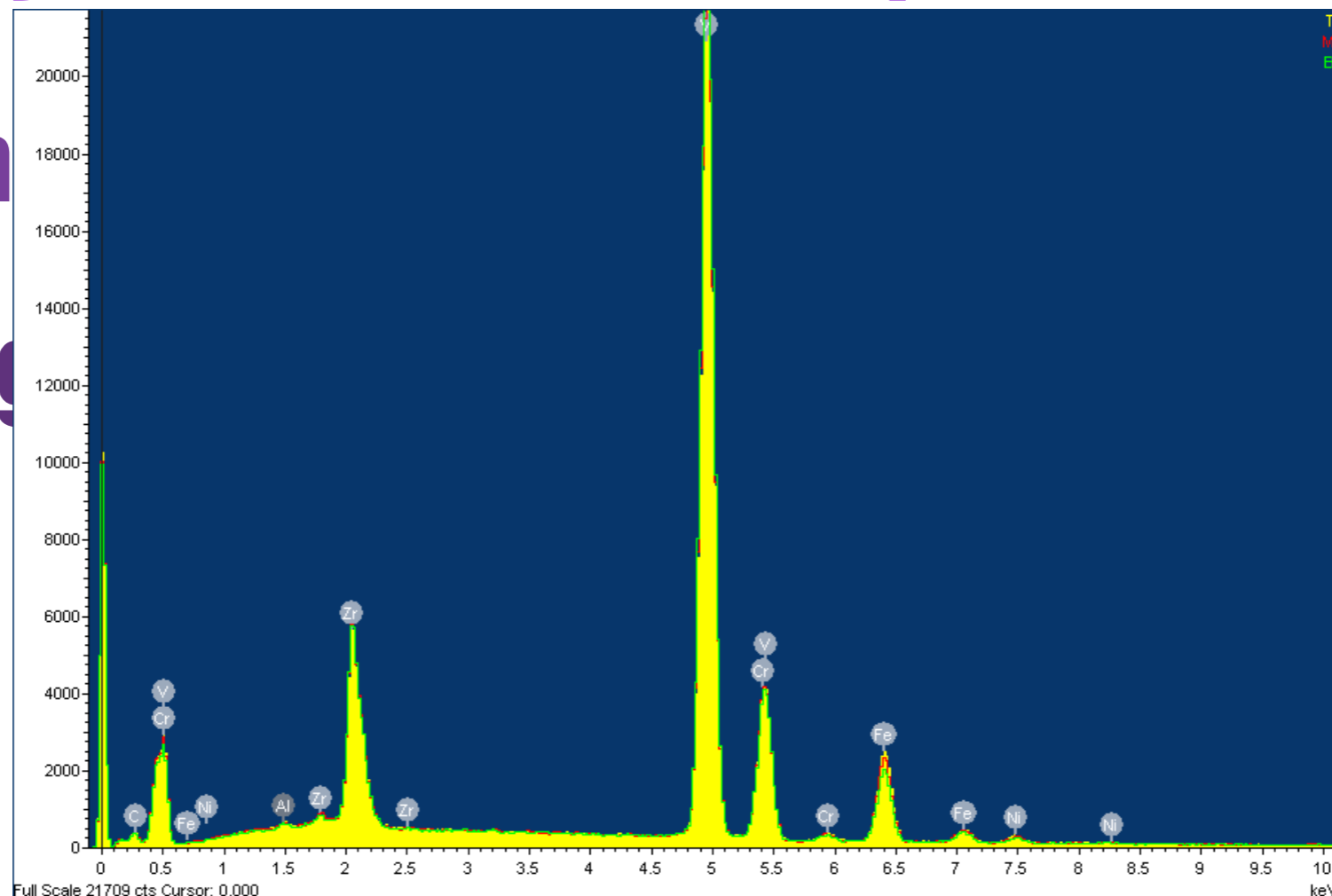


Microscopic Measurement

- X-Rays Photoelectron Spectroscopy - XPS

- Scanning Electron Microscopy - SEM

- Energy Dispersive X-ray Spectroscopy - EDS



Characterization Measurements

Coating Campaign



Microscopic Measurement

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

Characterization Measurements



Microscopic Measurement

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

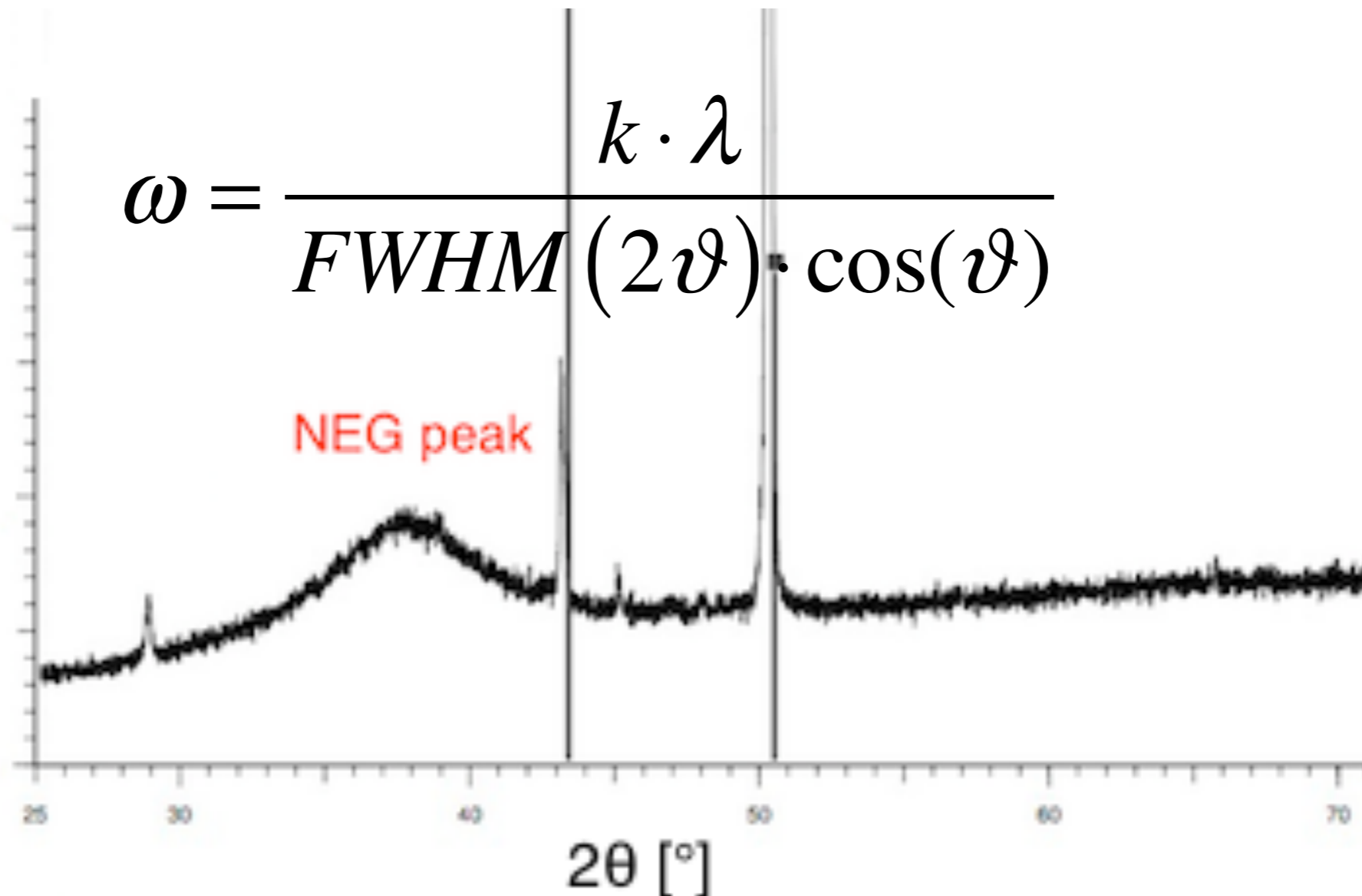
Characterization Measurements

Coating Campaign



Microscopic Measurement

- X
- S
- E
- >



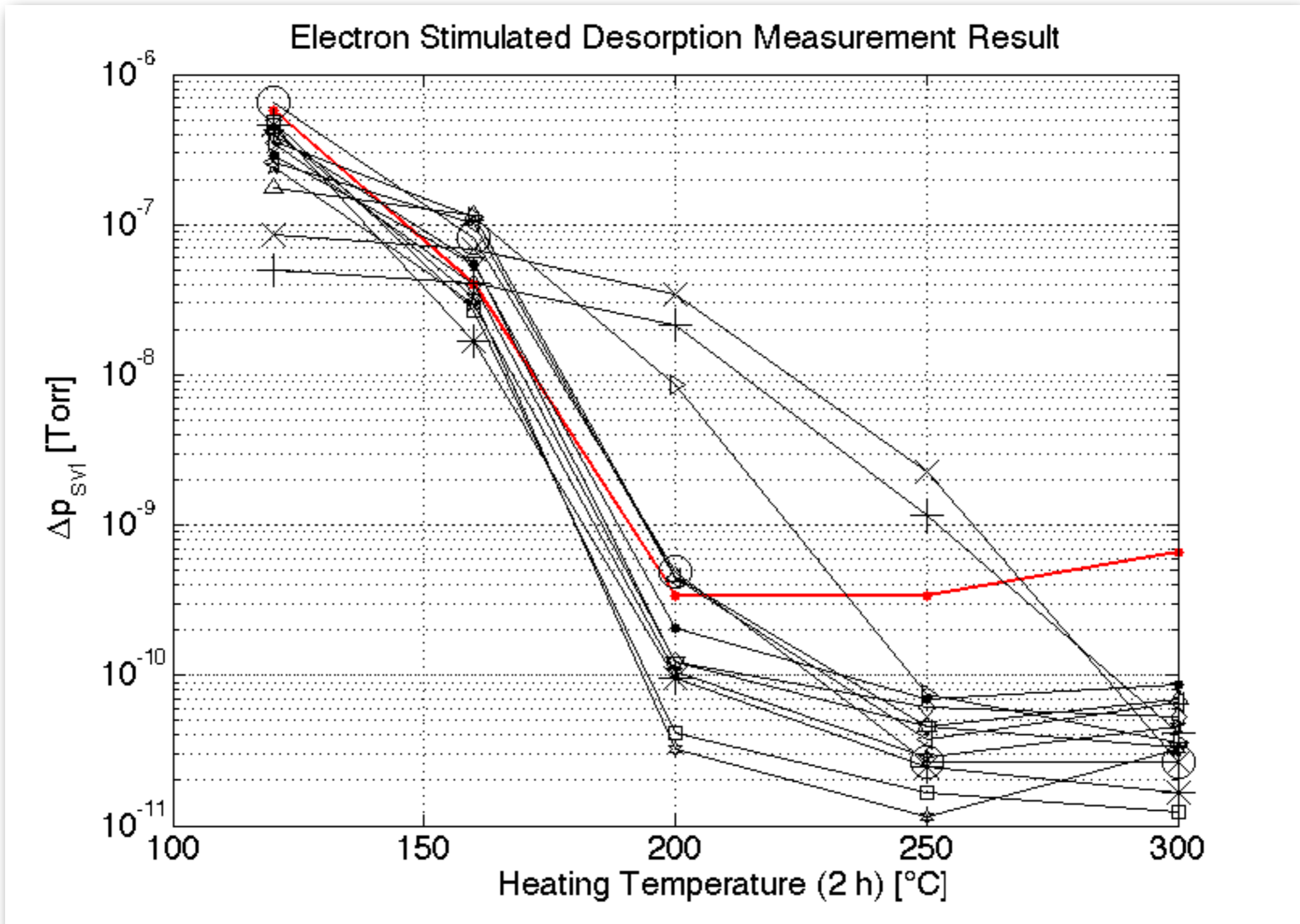
● XPS

● S

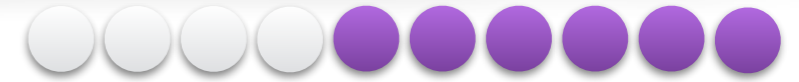
● RD

Coating Campaign - ZrV

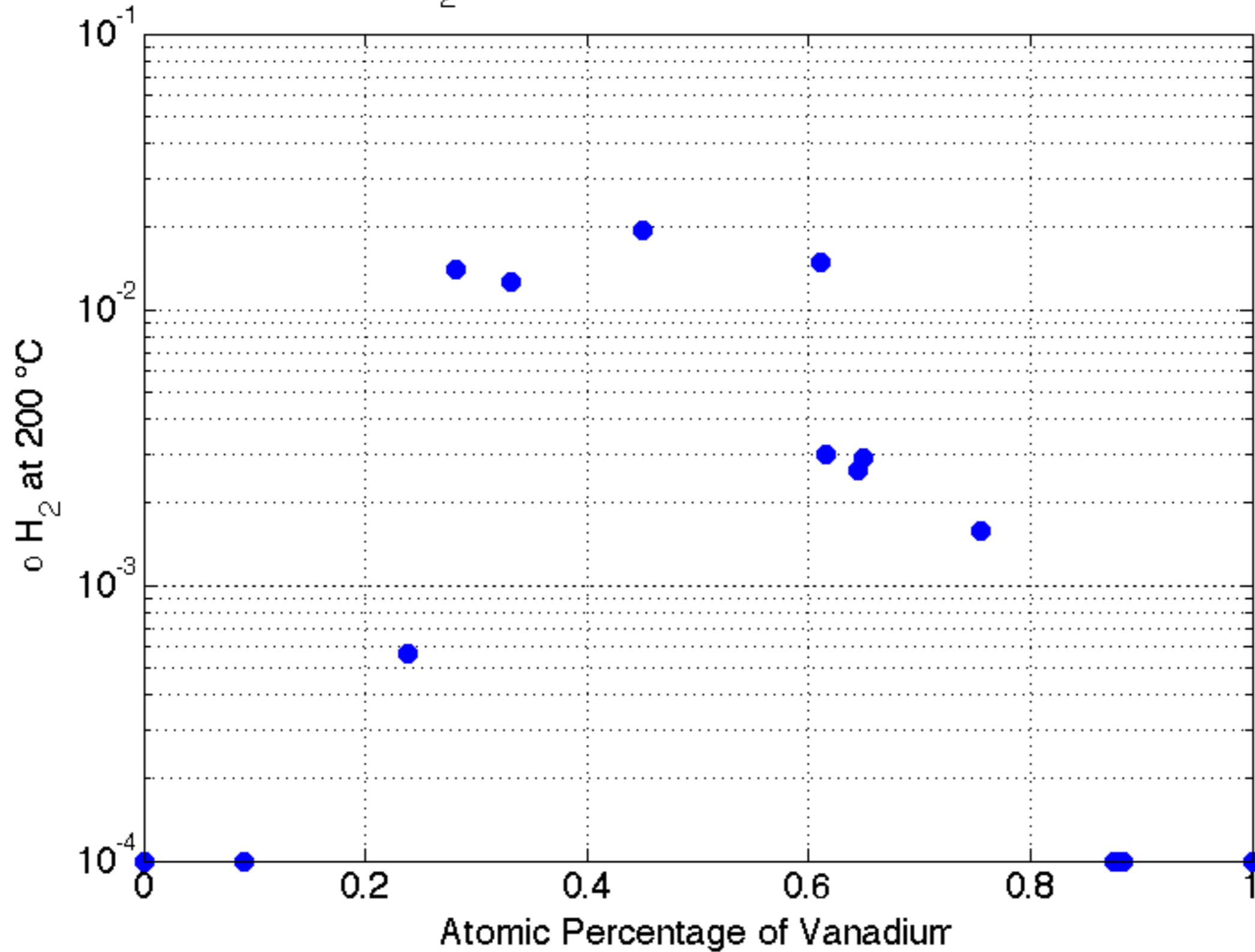
Coating Campaign - ZrV



Coating Campaign - ZrV



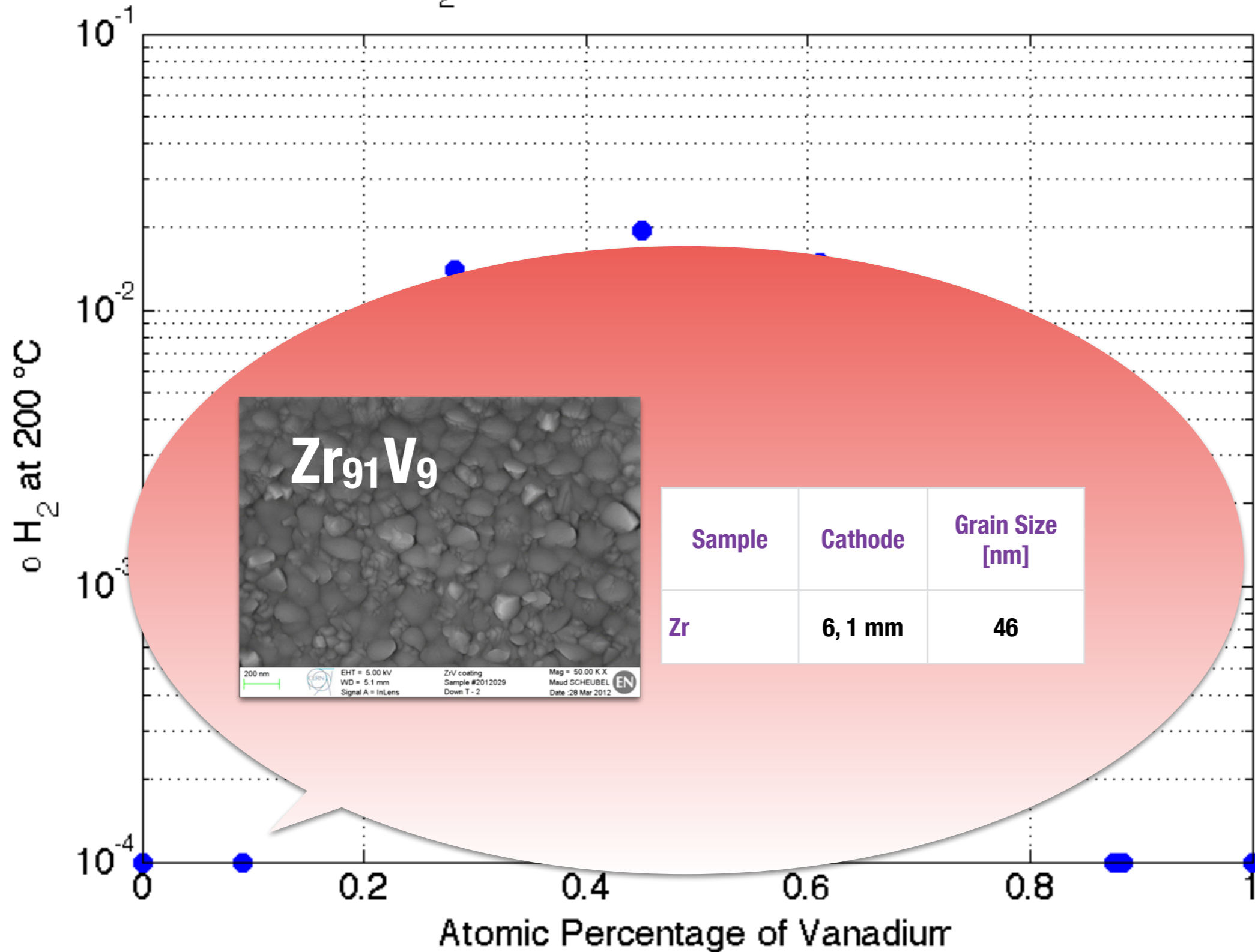
H₂ Sticking Probability at 200 °C



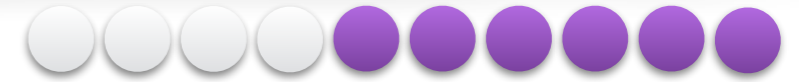
Coating Campaign - ZrV



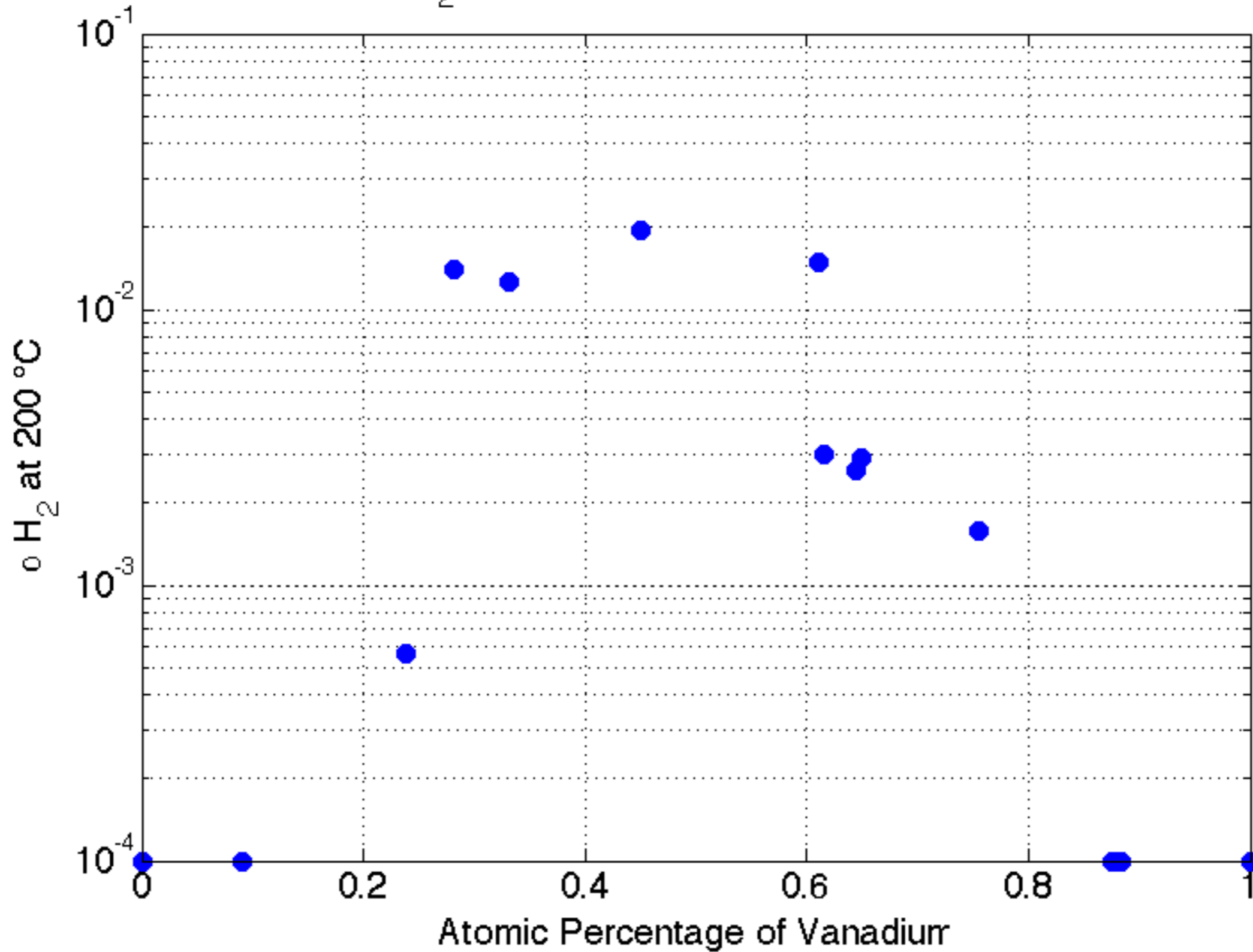
H₂ Sticking Probability at 200 °C



Coating Campaign - ZrV



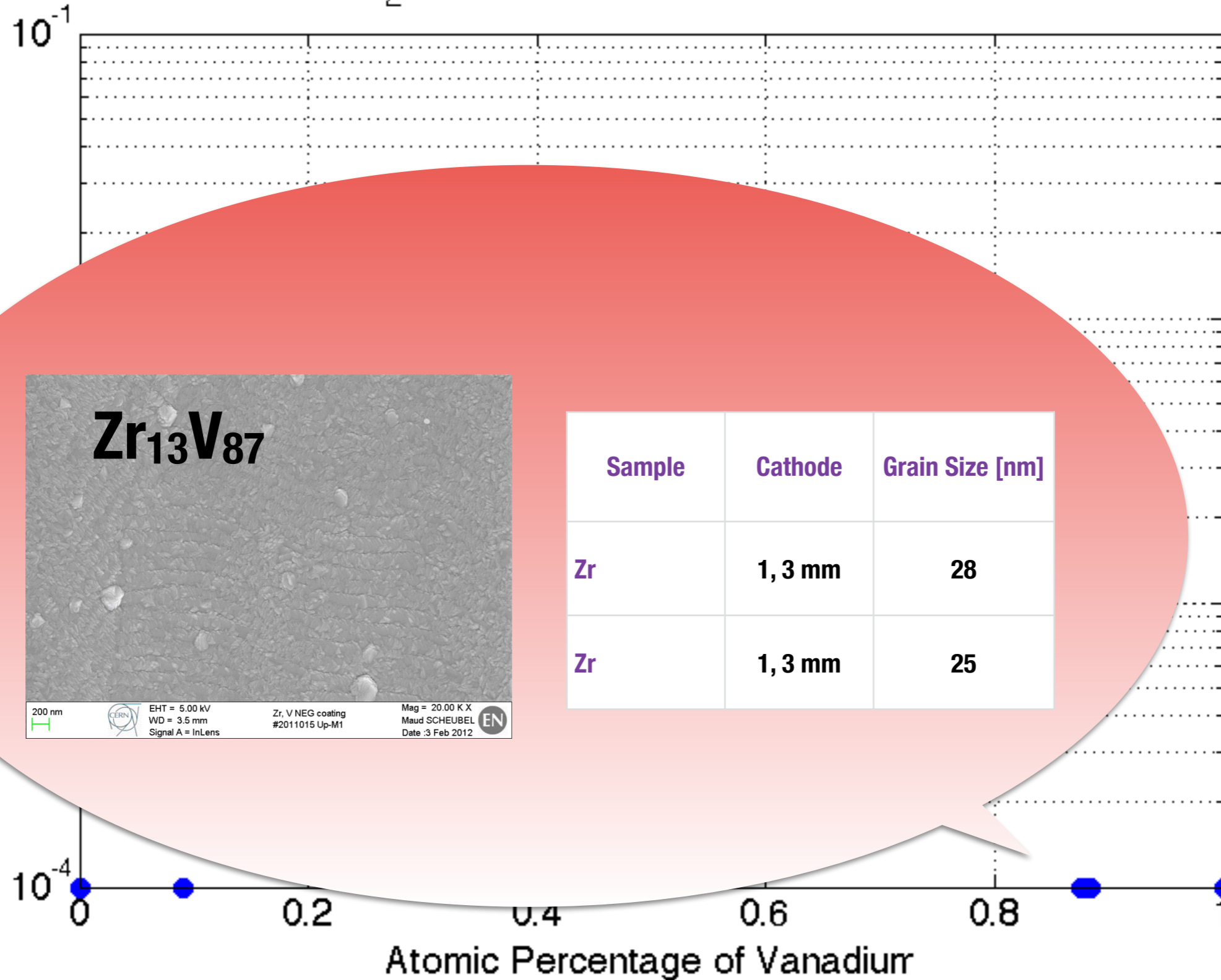
H₂ Sticking Probability at 200 °C



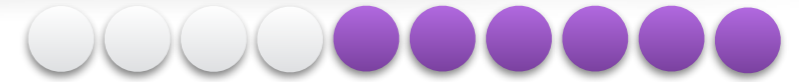
Coating Campaign - ZrV



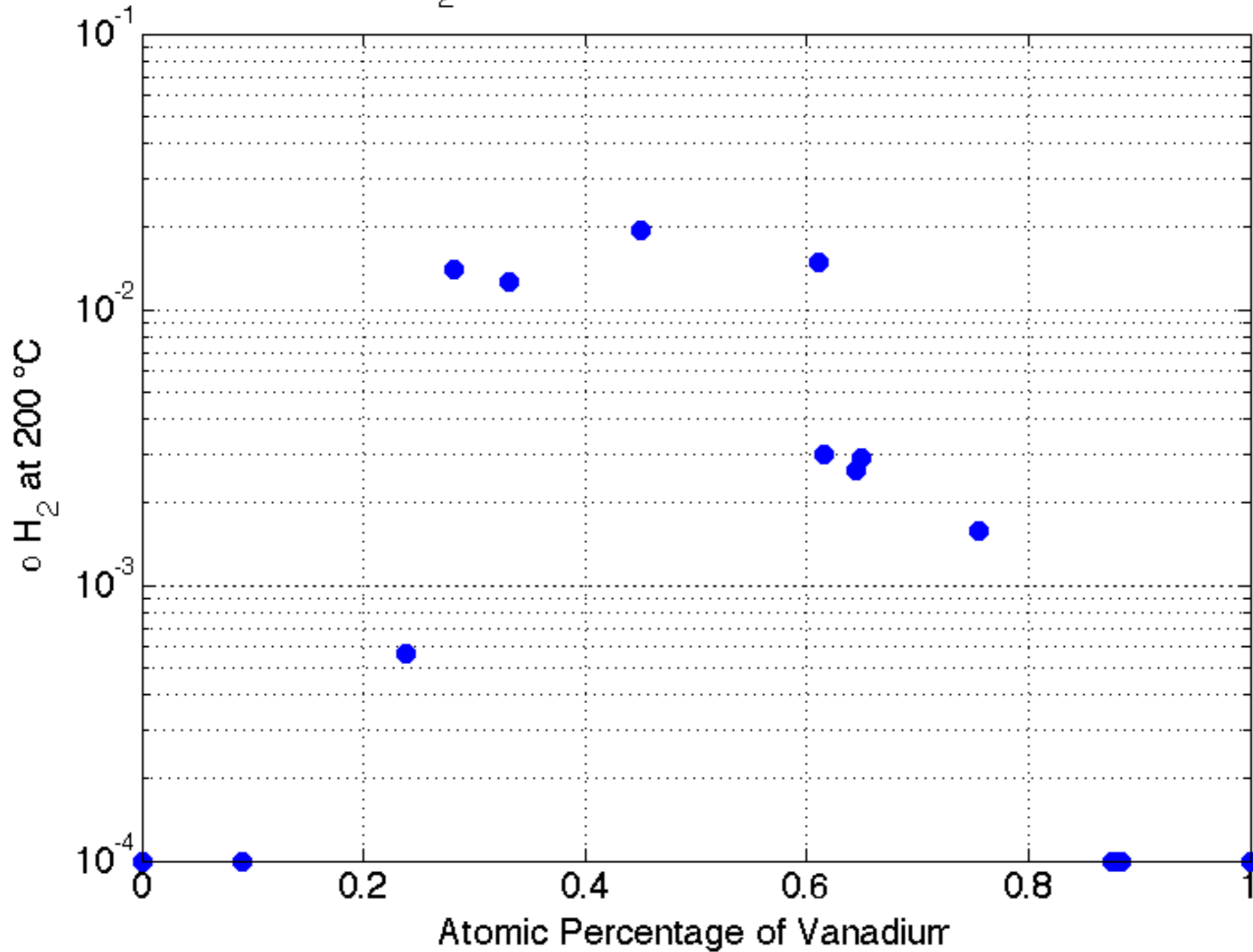
H₂ Sticking Probability at 200 °C



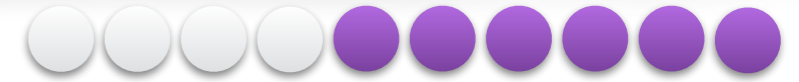
Coating Campaign - ZrV



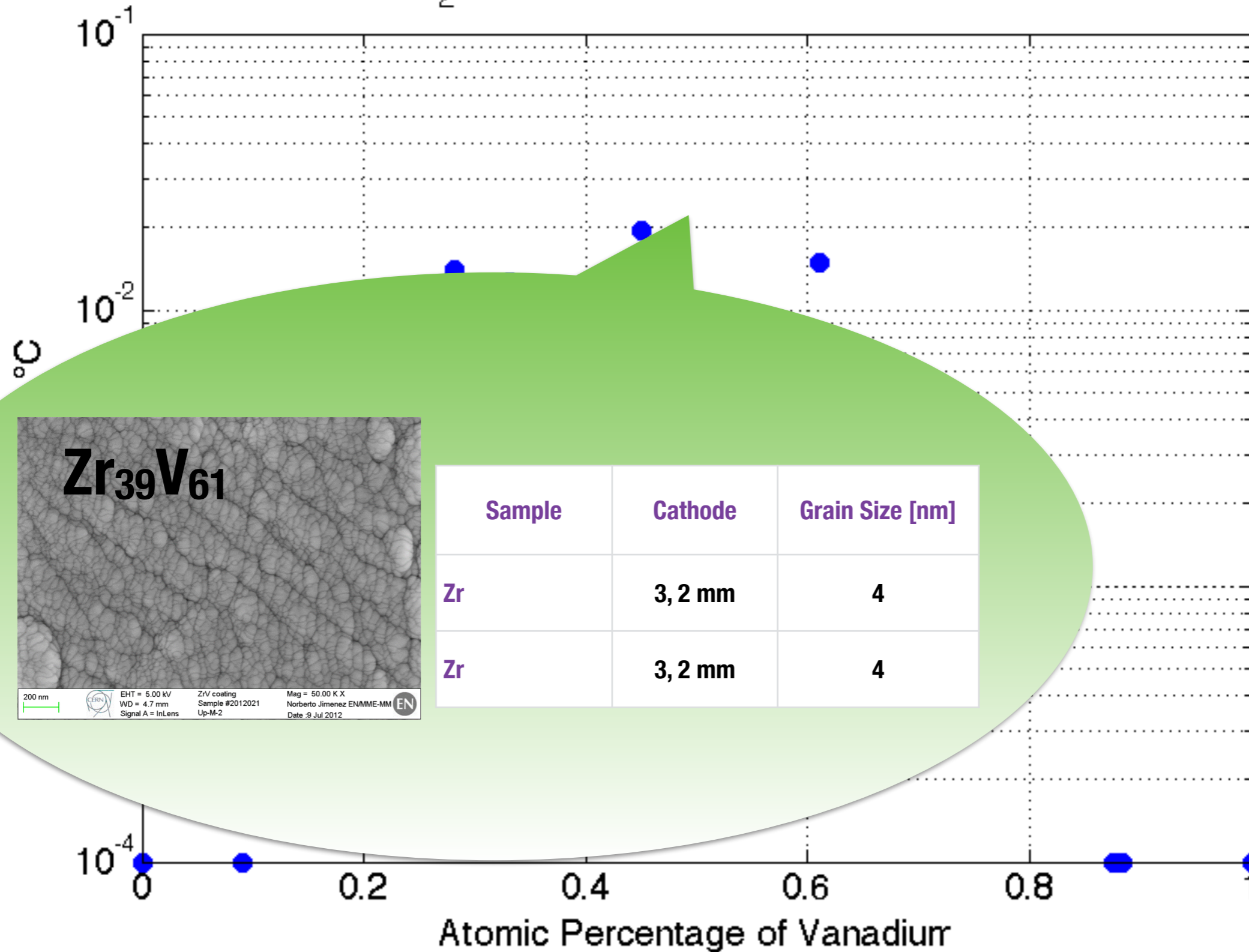
H₂ Sticking Probability at 200 °C



Coating Campaign - ZrV



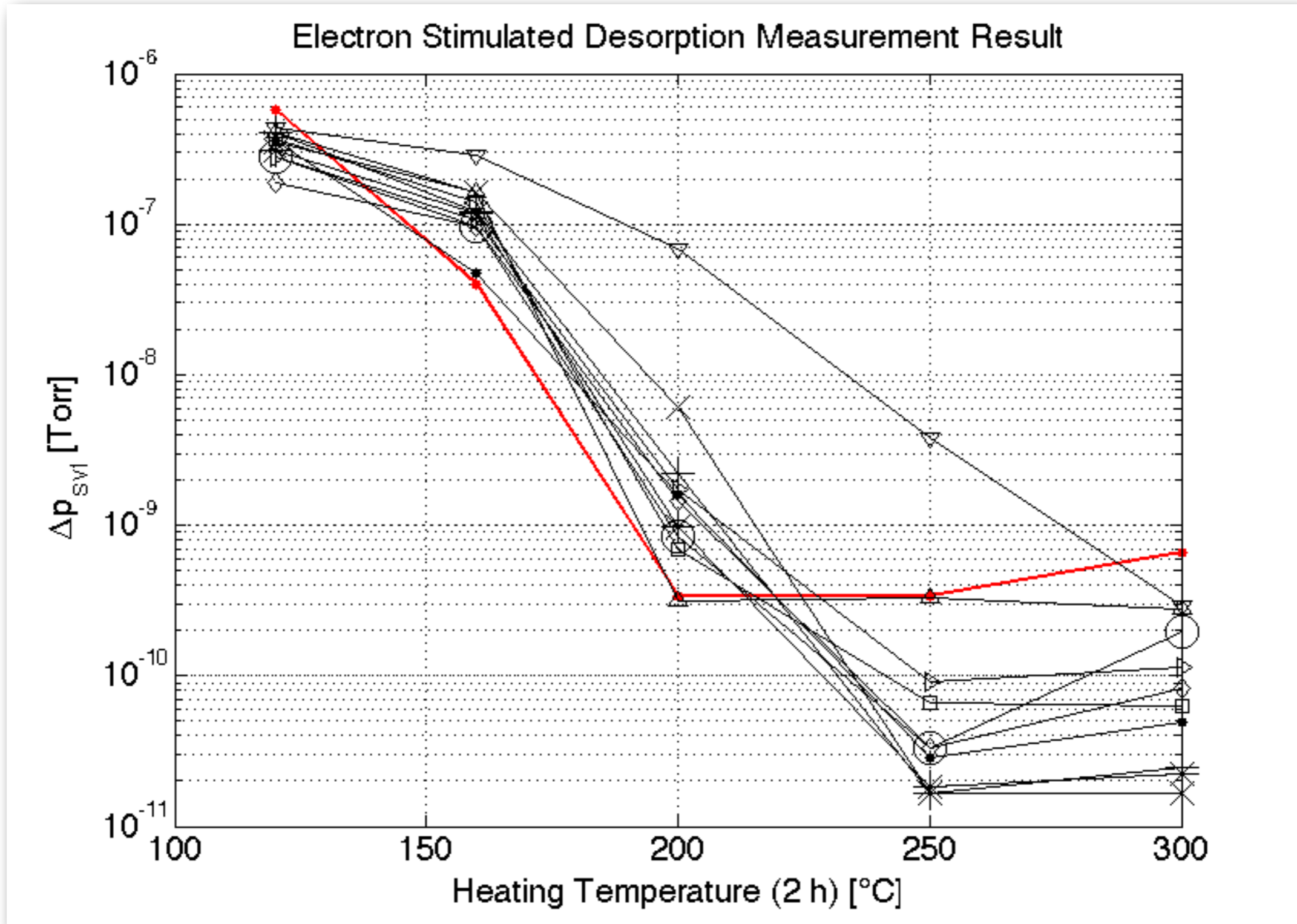
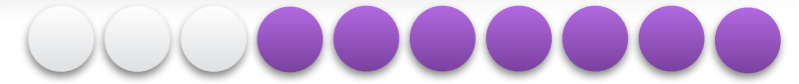
H₂ Sticking Probability at 200 °C



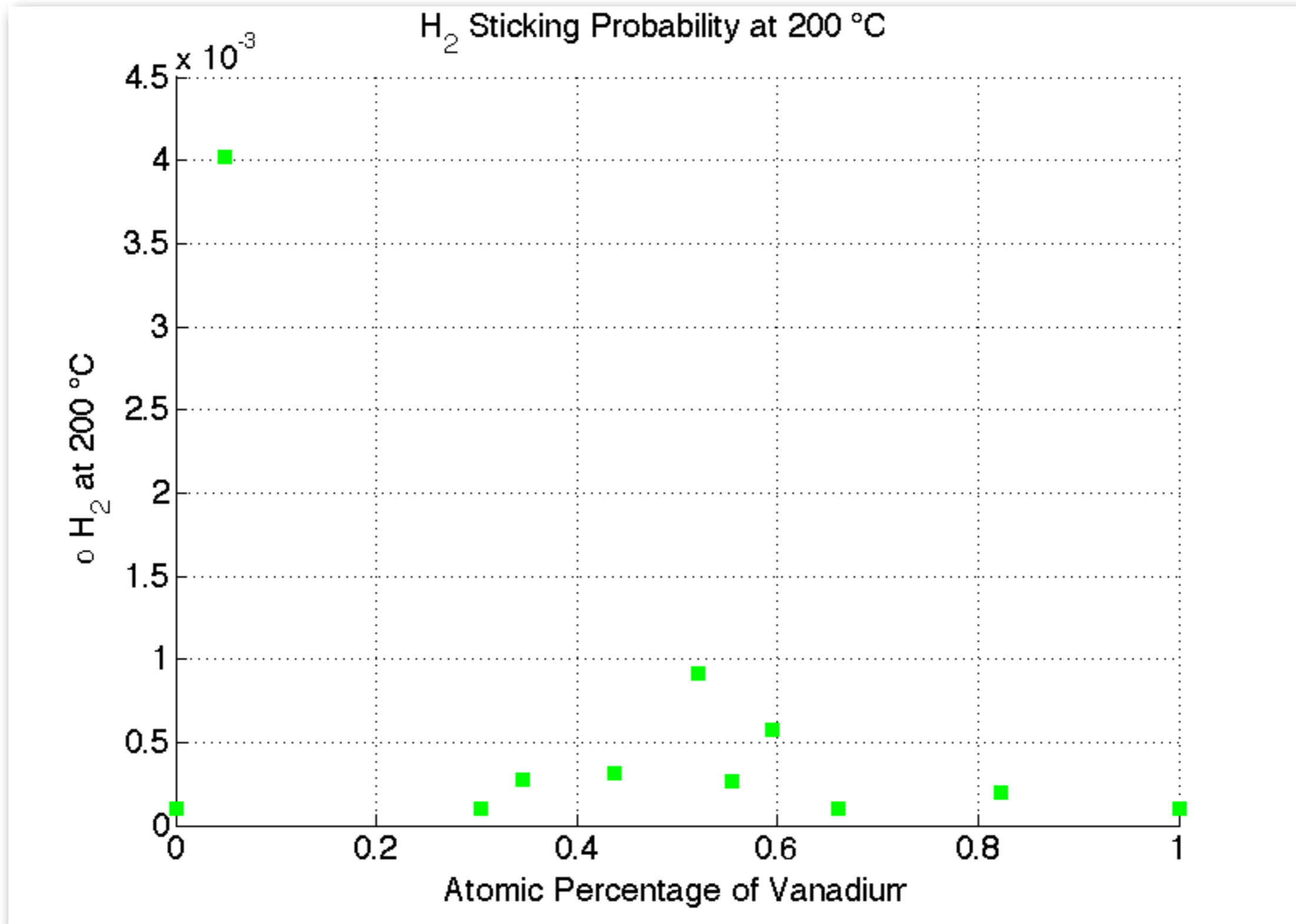
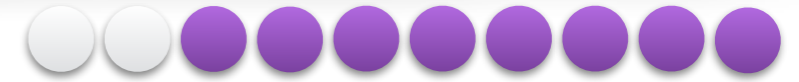
Coating Campaign - HfV

Experimental Results

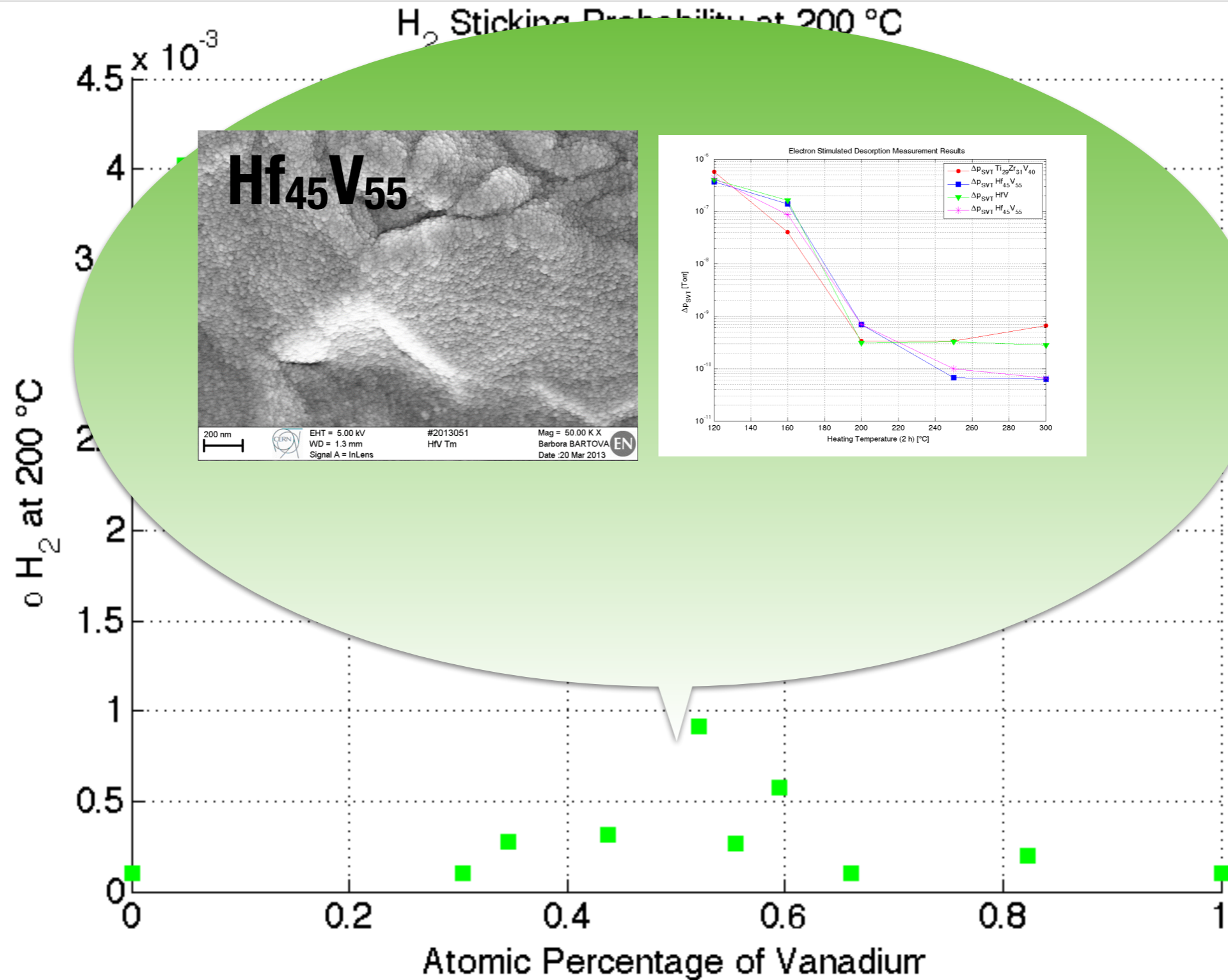
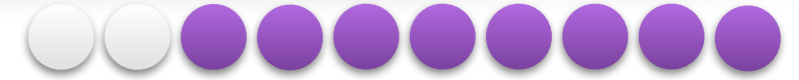
Coating Campaign - HfV



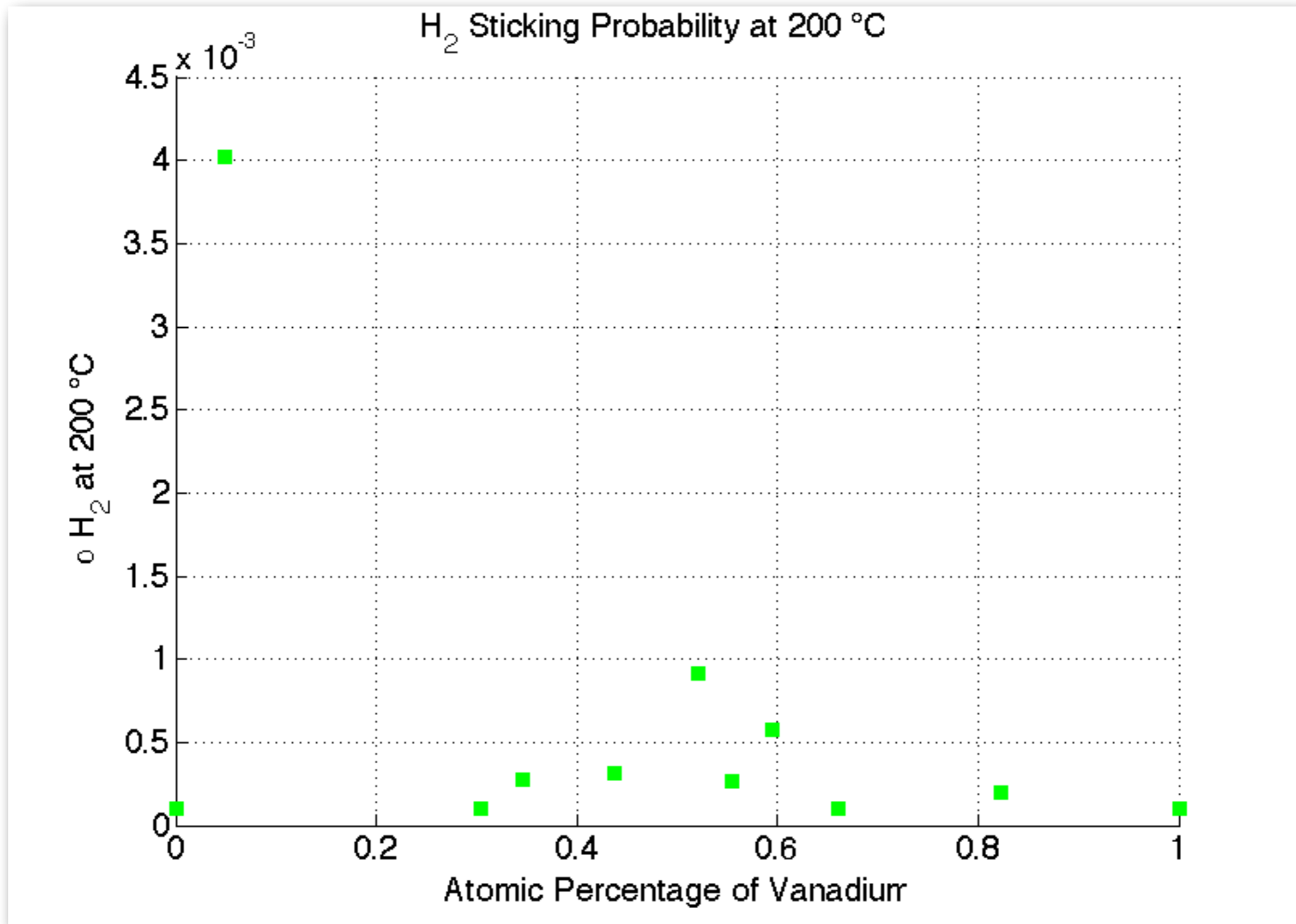
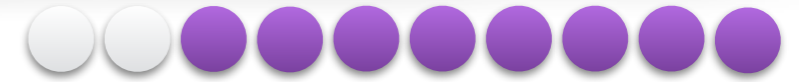
Coating Campaign - HfV



Coating Campaign - HfV

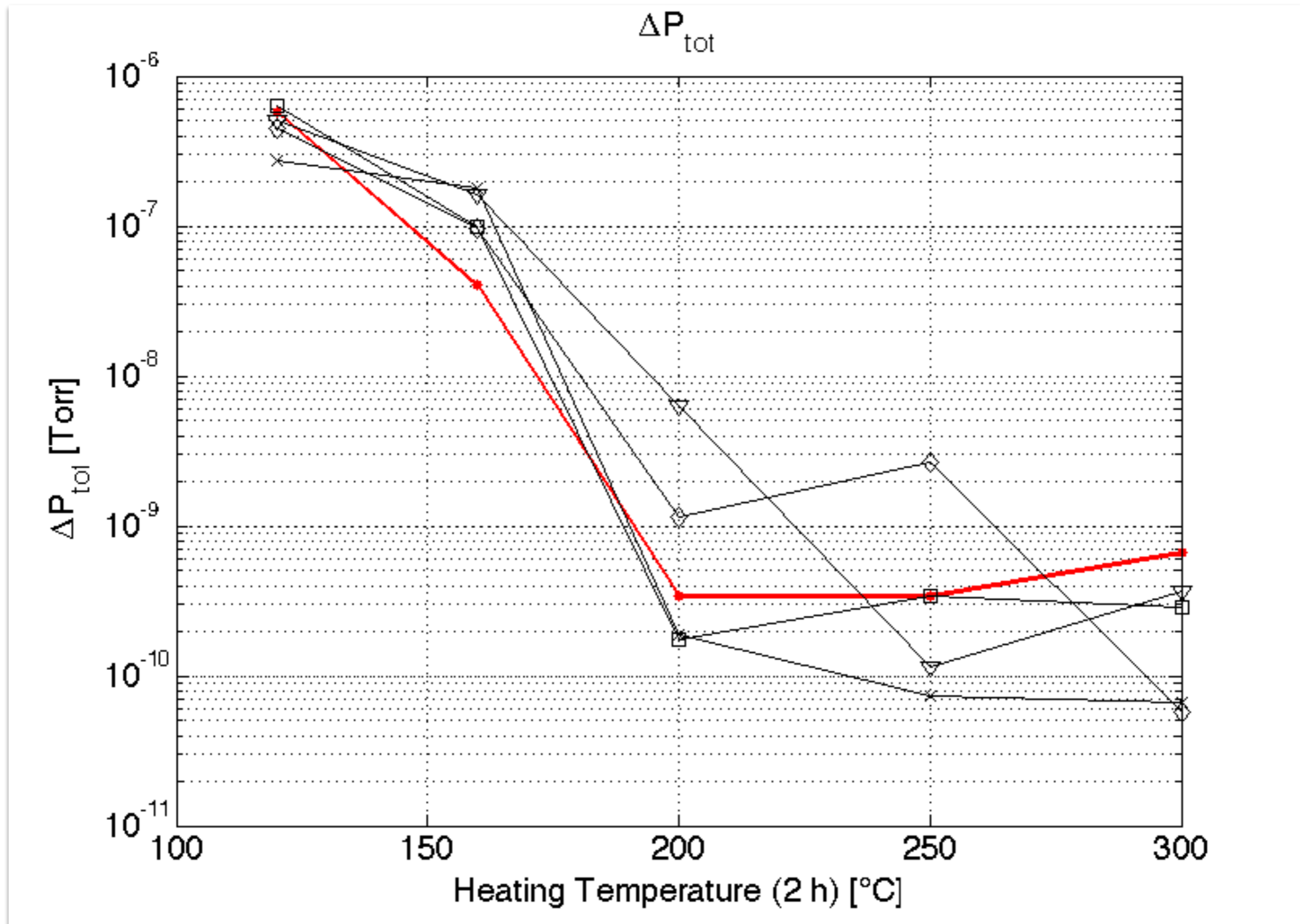


Coating Campaign - HfV

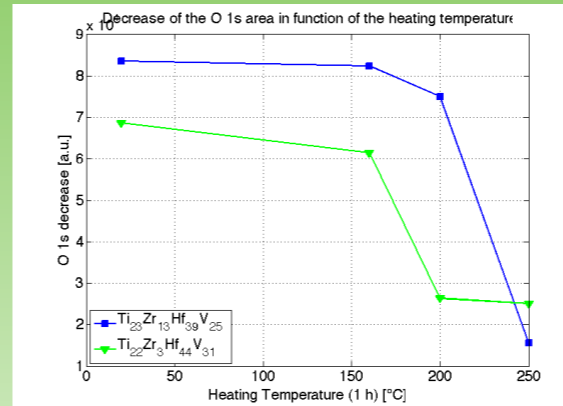
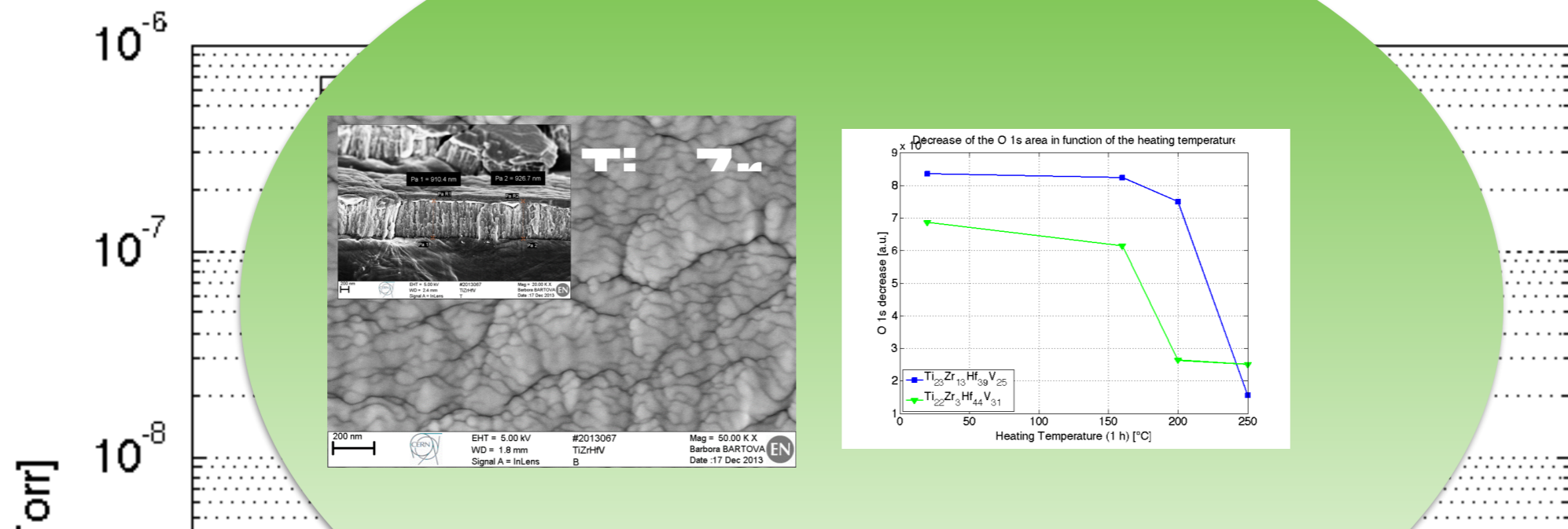


Coating Campaign - TiZrHfV

Coating Campaign - TiZrHfV



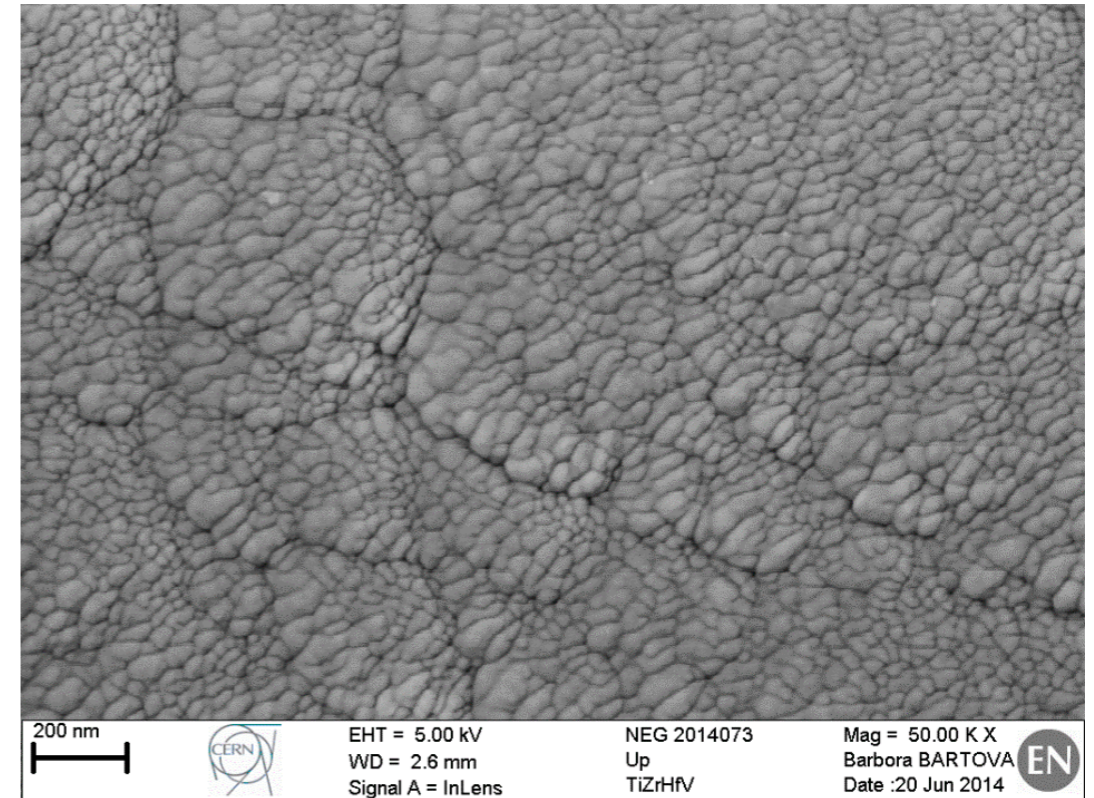
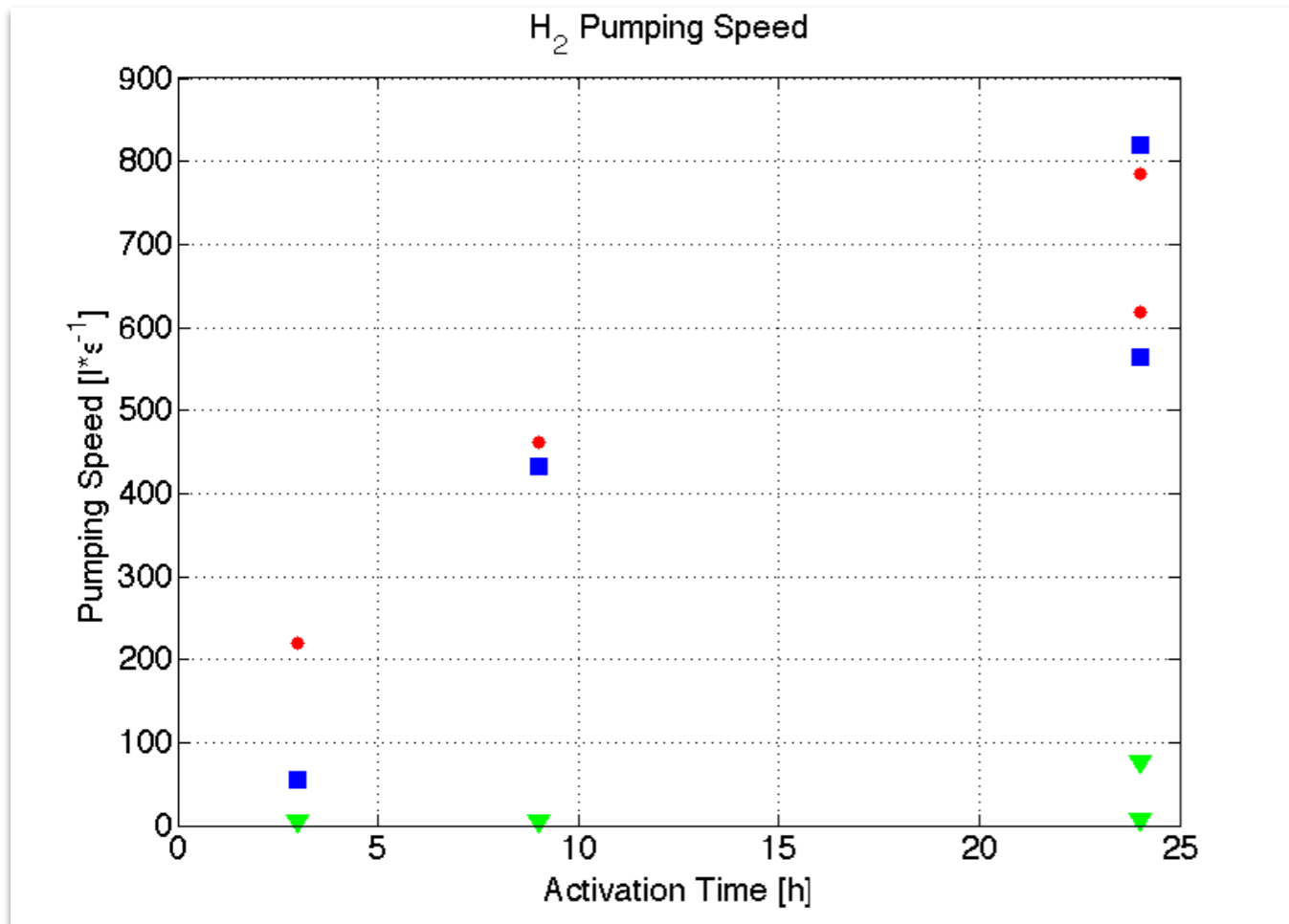
Coating Campaign - TiZrHfV



ΔP_{tot} [Torr]	Sample	Cathode	Grain Size [nm]
1	Ti	1,1,1,1 mm	2
1	Ti	2,1,2,2 mm	1

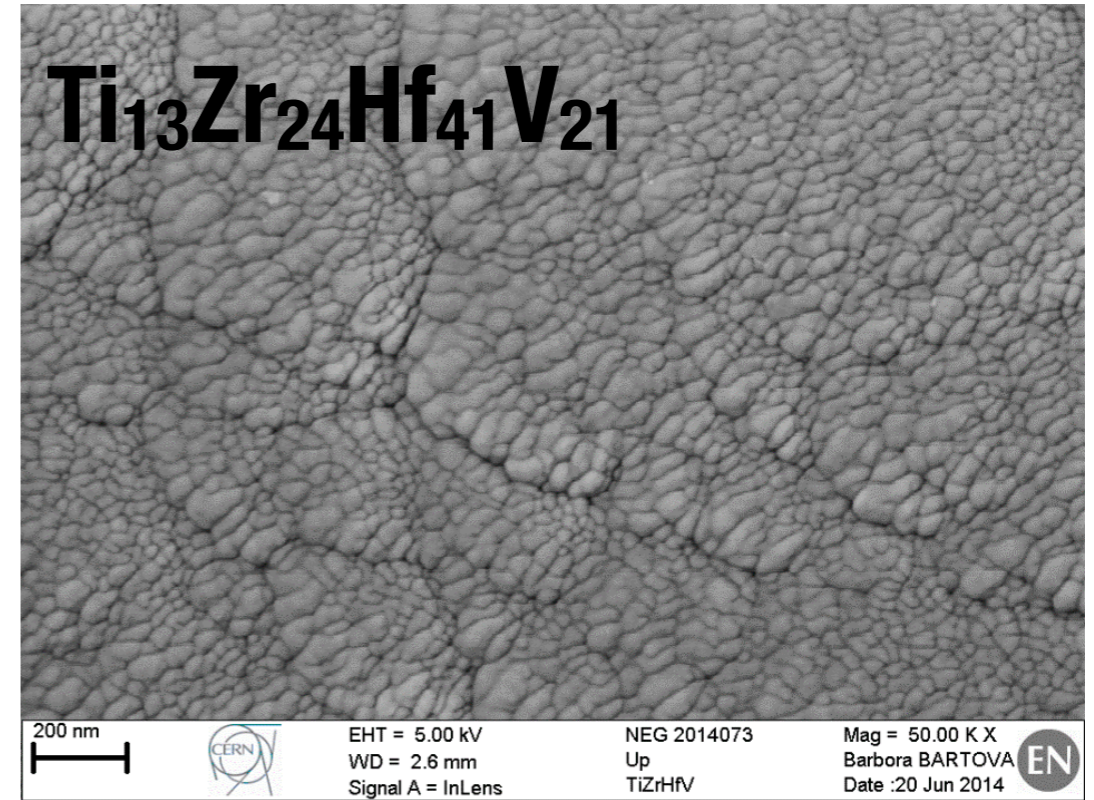
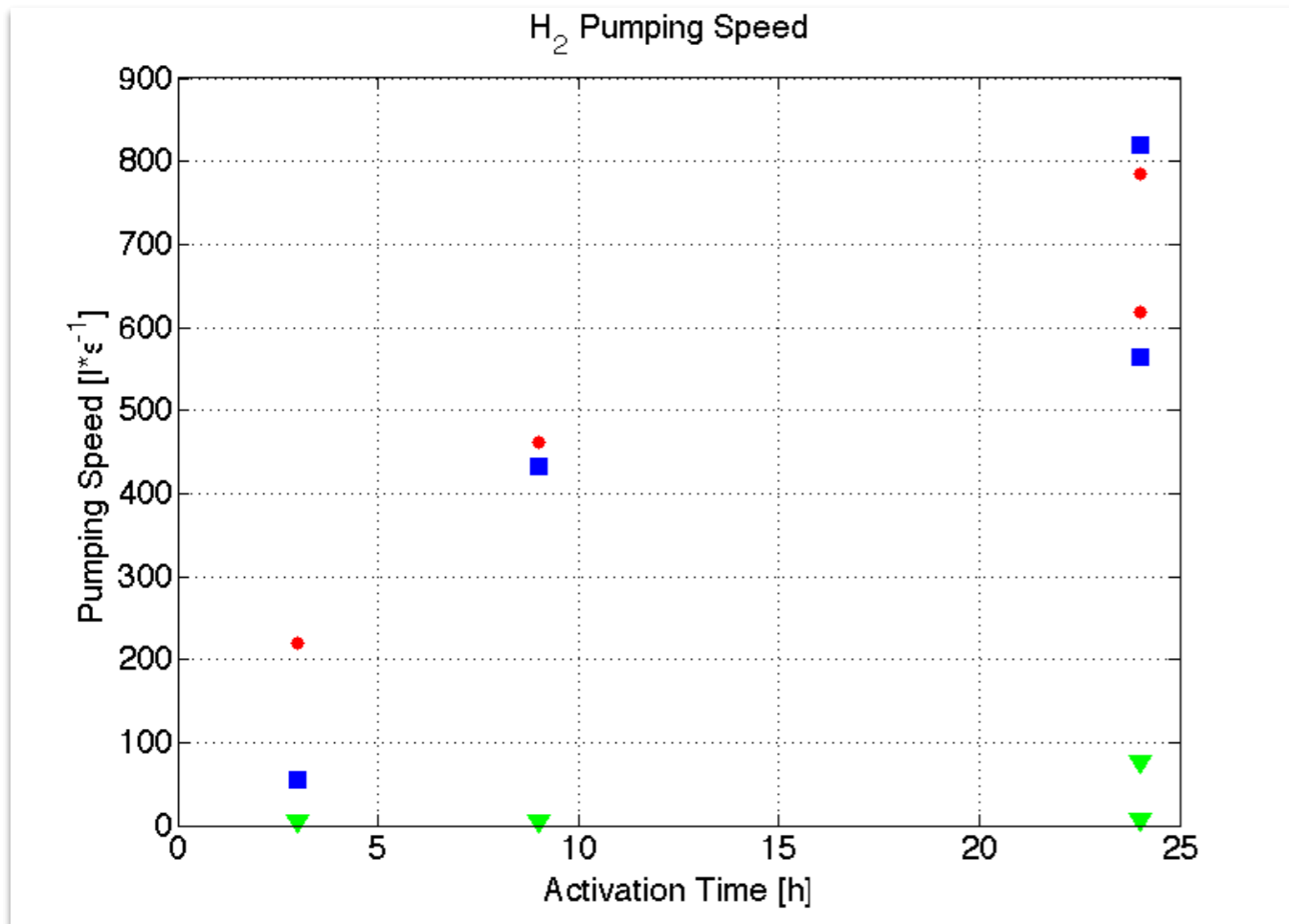
Heating Temperature (2 h) [°C]

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



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

Conclusions

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**



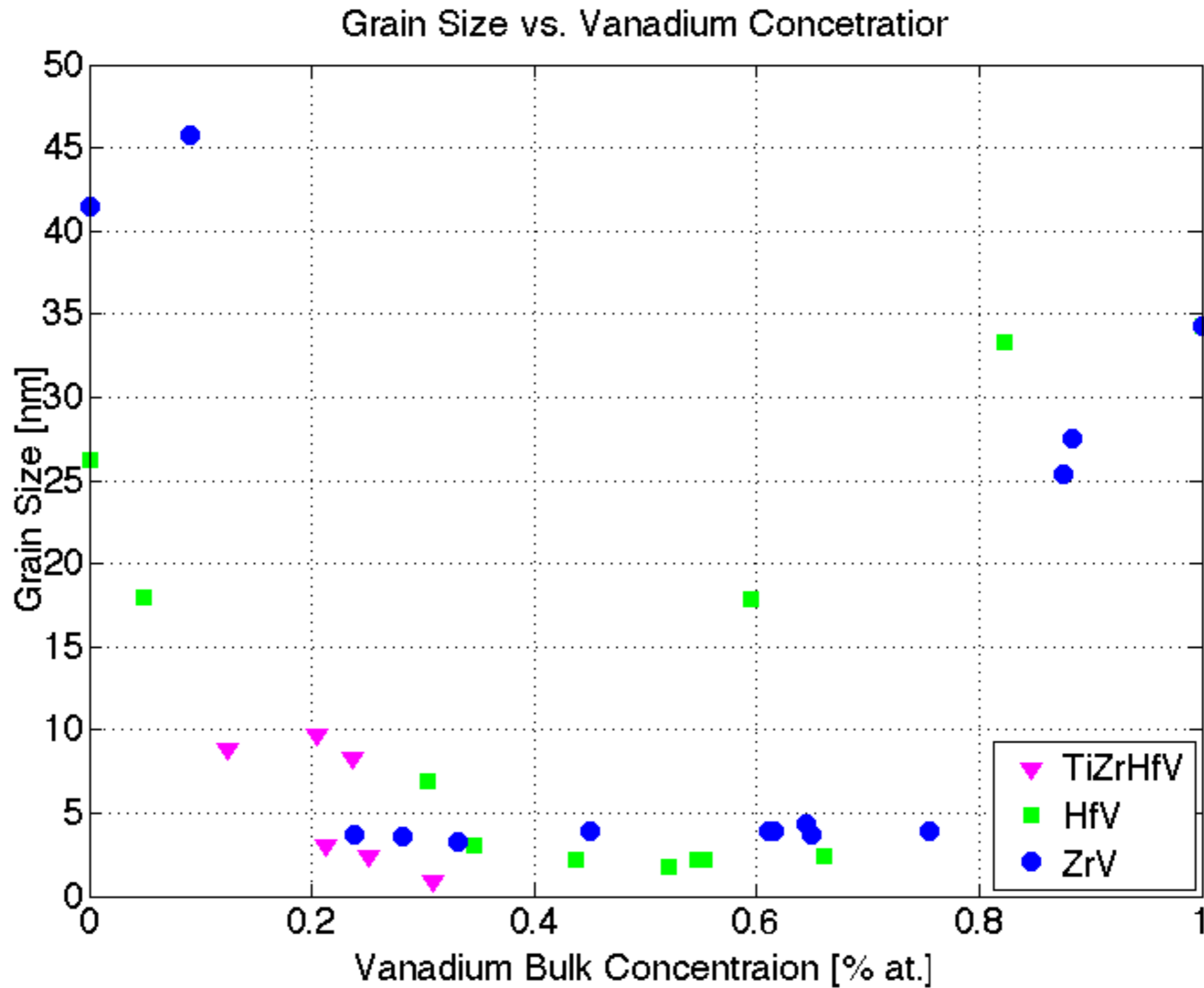
- **Ageing Campaign**

- **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**



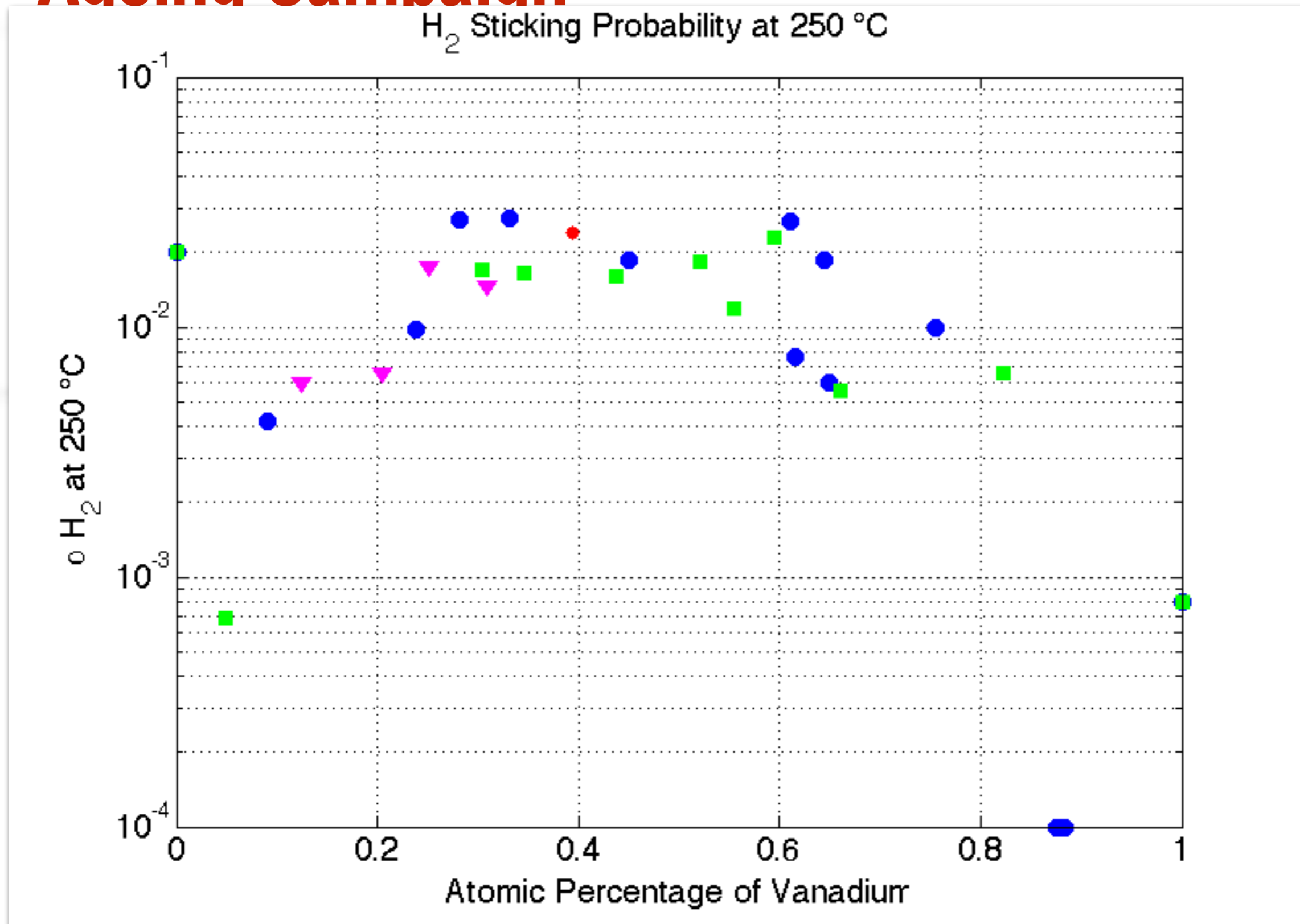
● Ageing Campaign

- **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