Characterization of deposited carbon layers in Tore Supra

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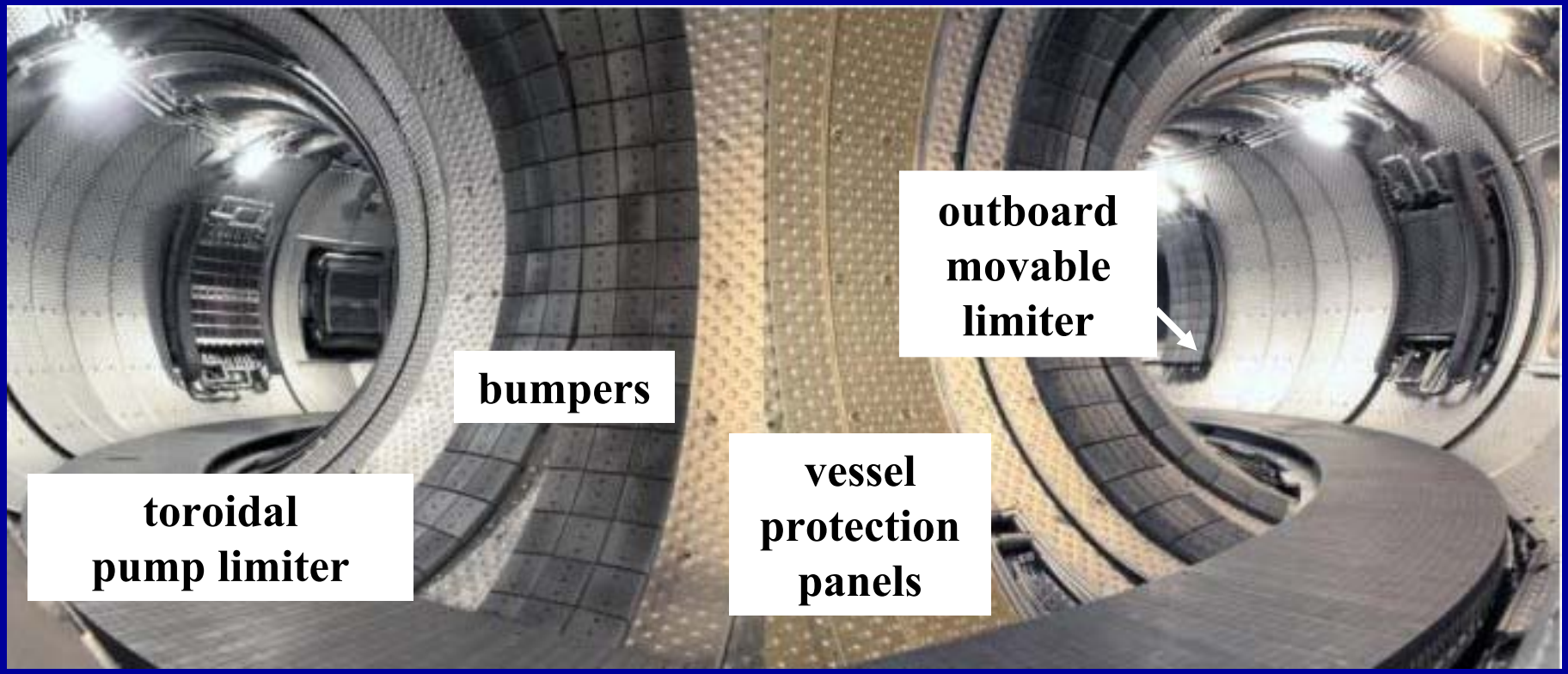
aims

- fuel balance in Tore Supra, contents in D

- multi-scale analysis of structure (atomic, nano, meso and macro)

  consequences on transport and diffusion properties

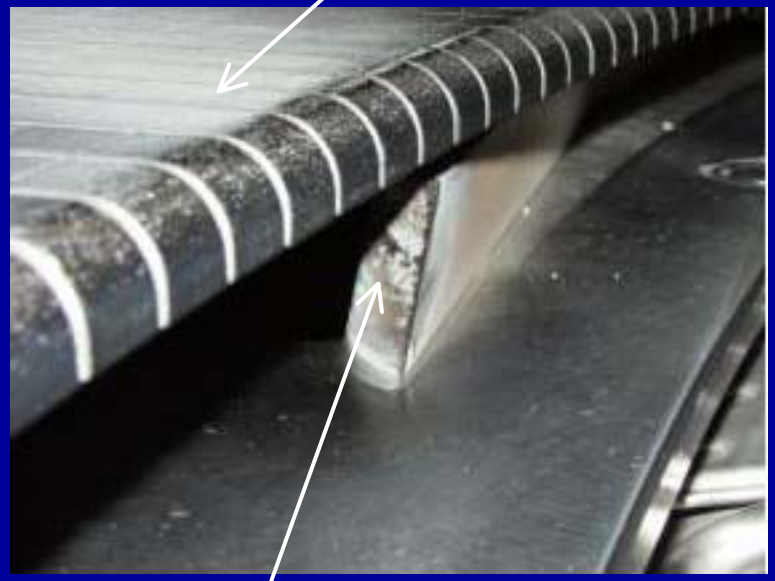
Tore Supra chamber



carbon - carbon composite (CFC): pyrolytic matrix - carbon fiber

$T < 450\ °C$
$\Phi(D^+) = 10^{17}\ cm^{-2}\ s^{-1}$
$E_i = 500\ eV$

limiter deposits: **LIM**



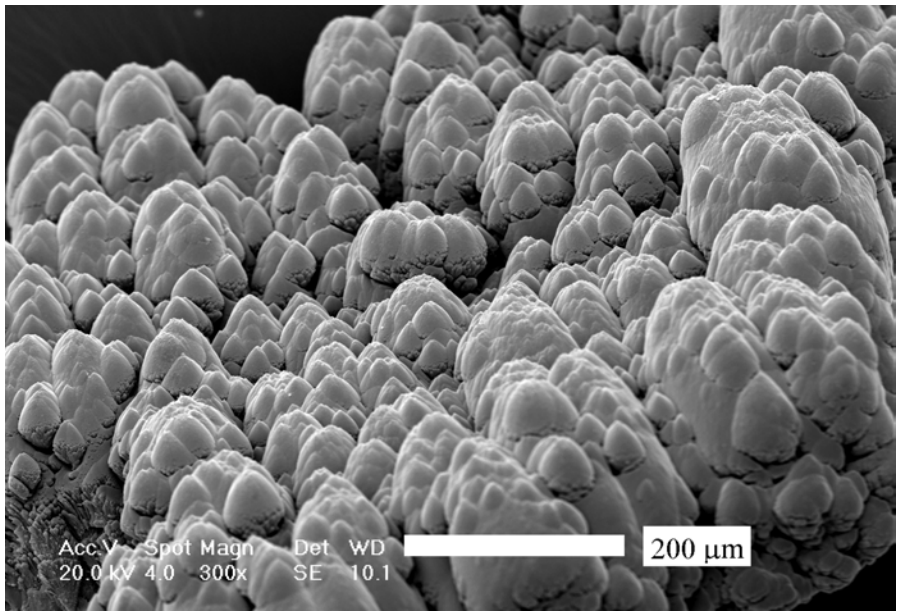
neutraliser deposits: **NTR**

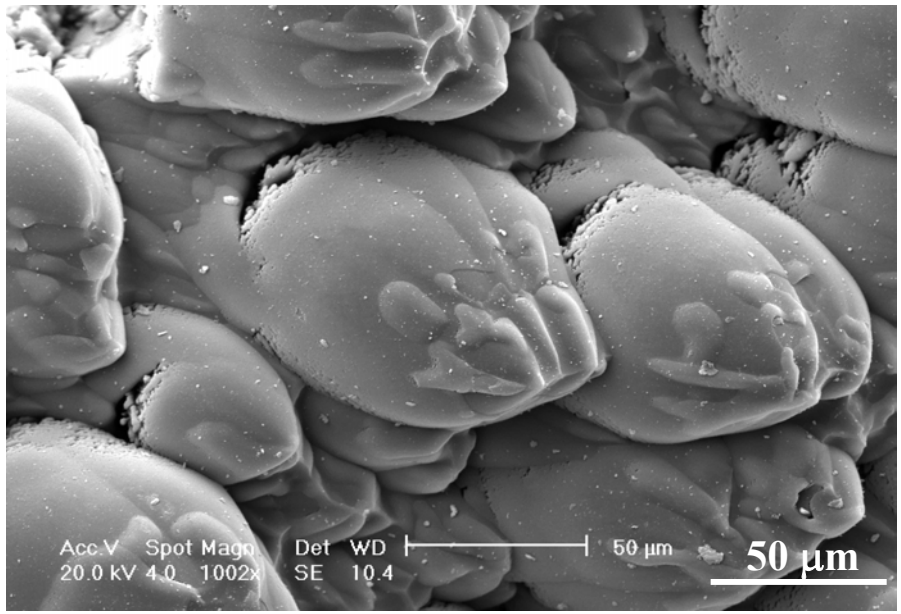
$T \sim 900\ °C$ (up to $1200\ °C$)
$\Phi(D^+) = 10^{16} - 10^{17}\ cm^{-2}\ s^{-1}$
$E_i = 100\ eV$

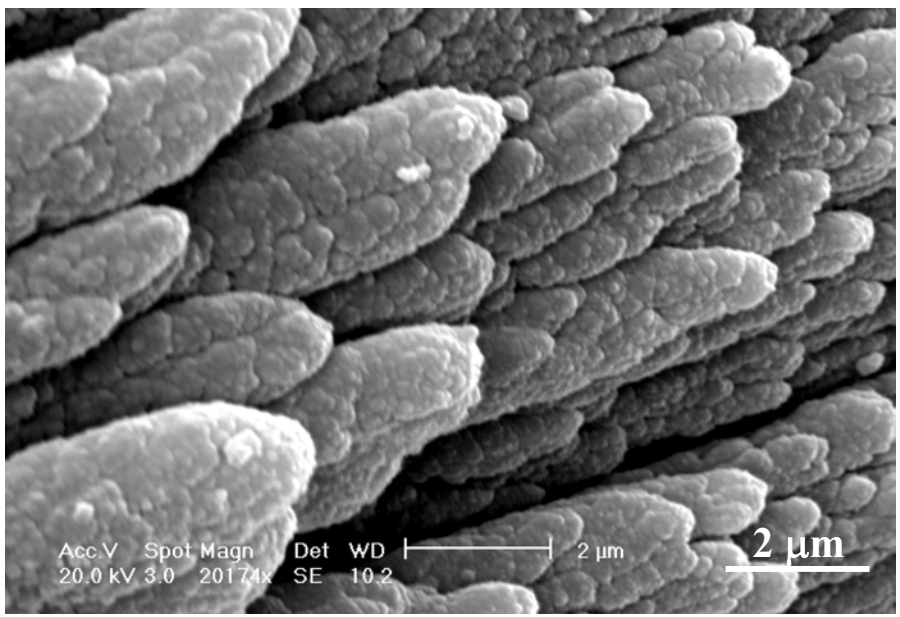
vertical outboard limiter: VOL

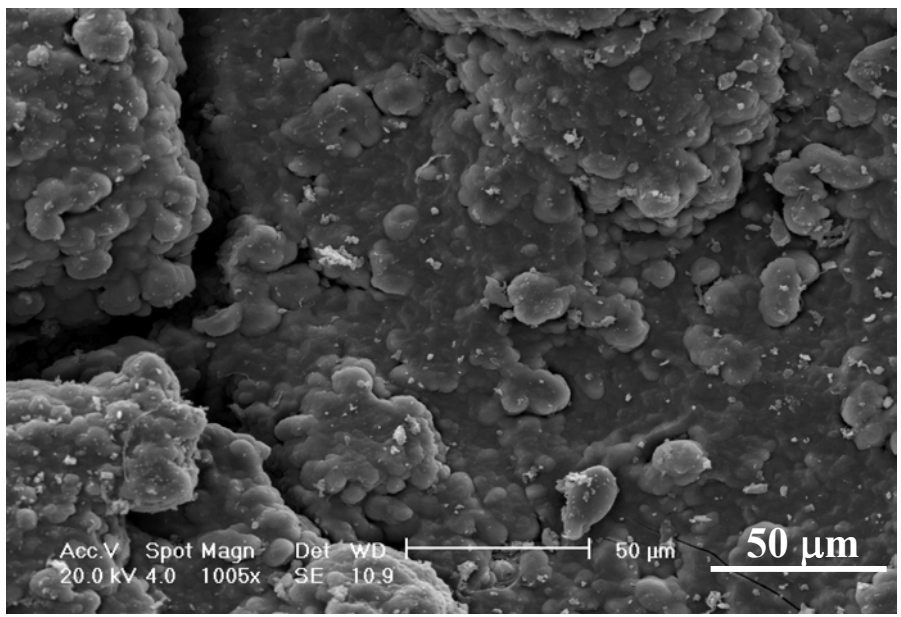
SEM: NTR deposits

fractal dimension: 2.15

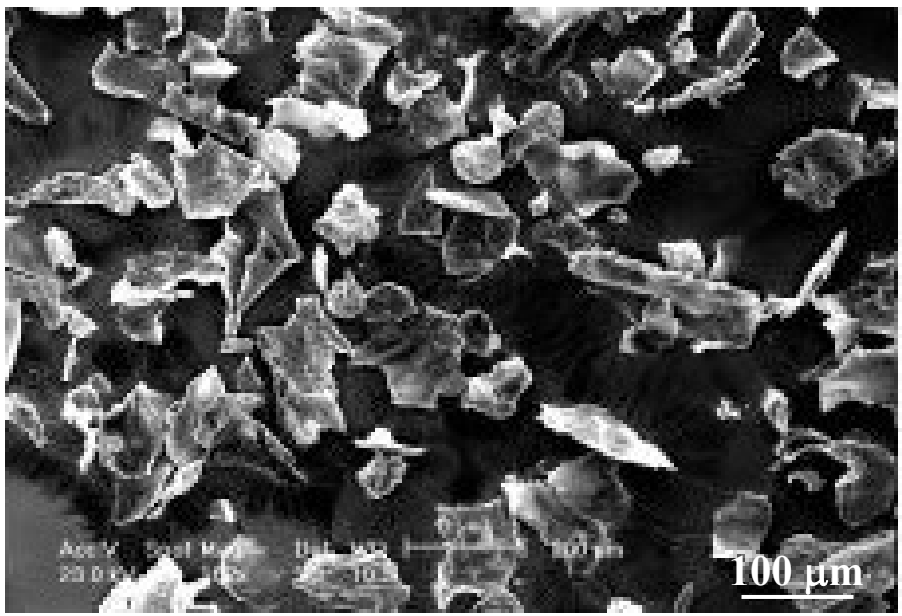


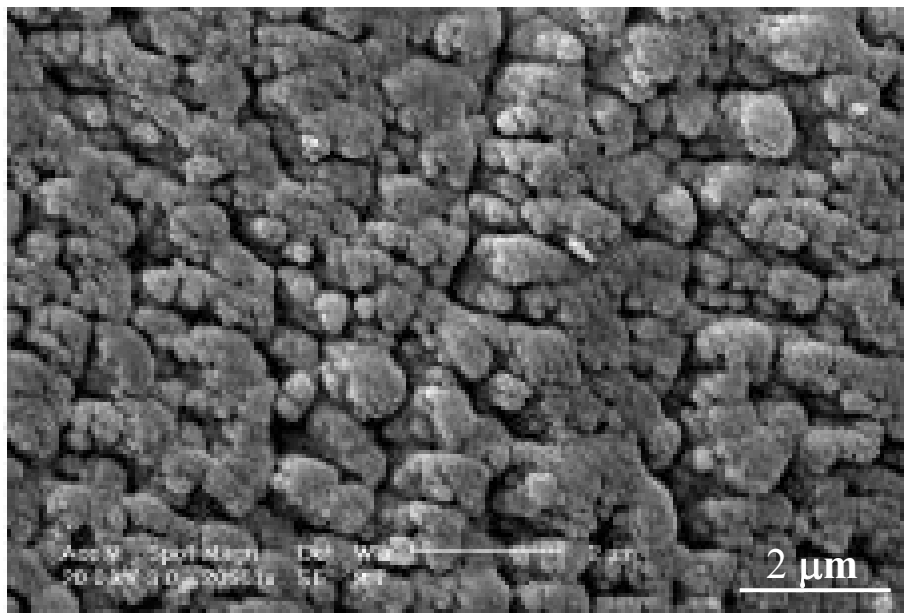






SEM: LIM deposits

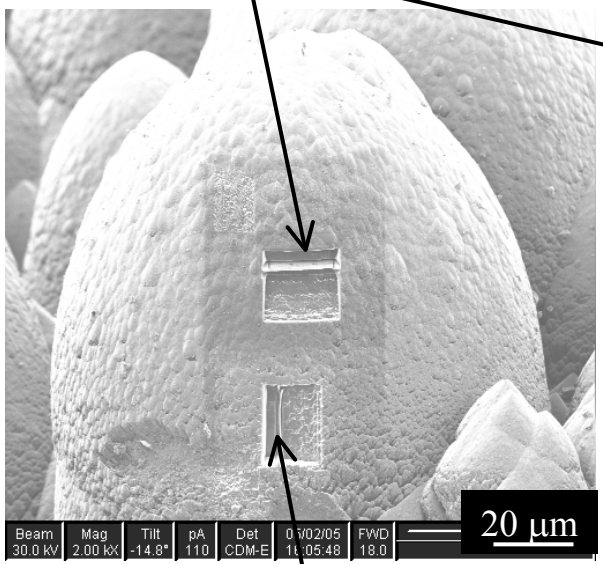


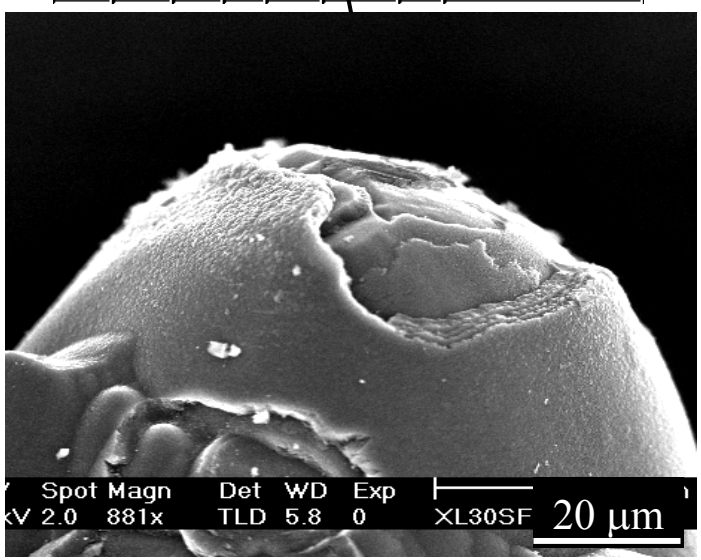


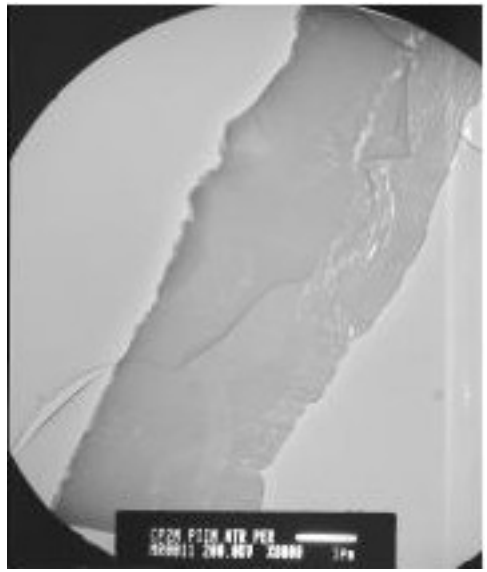
TEM: NTR

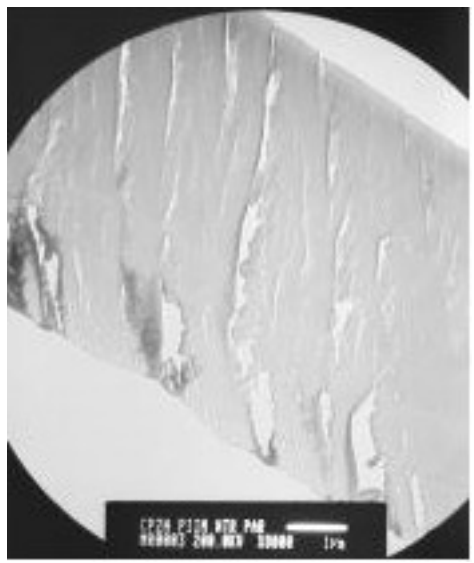
FIB

perpendicular foil

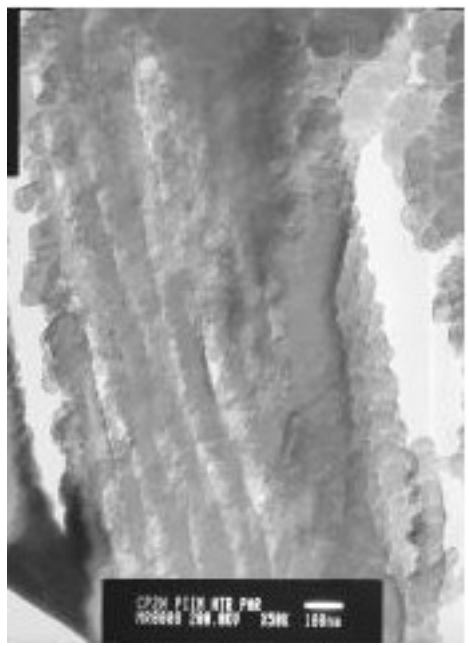






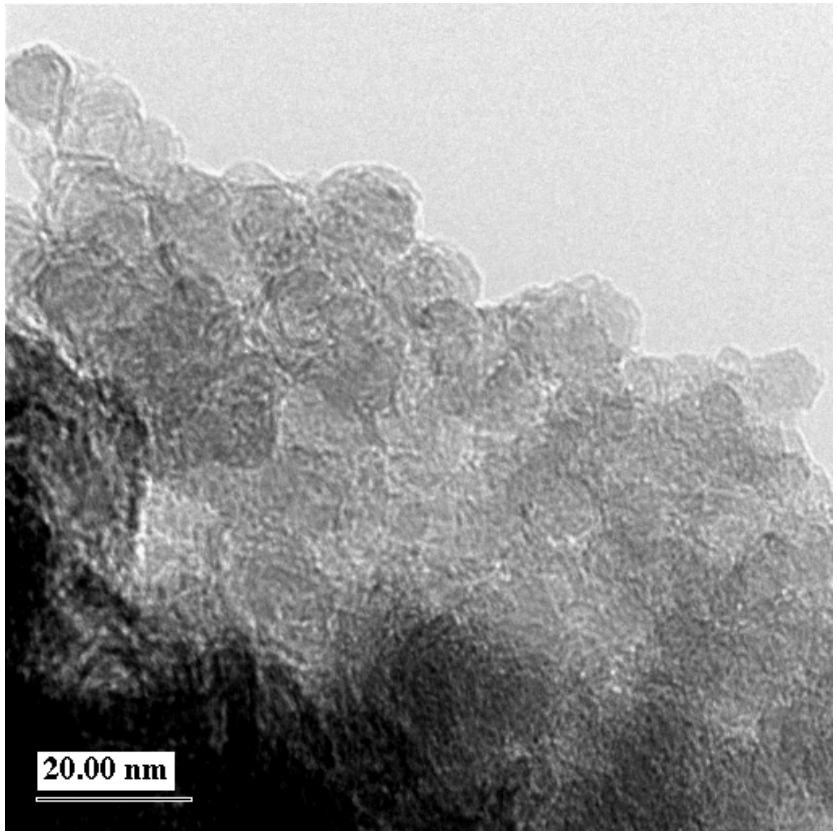


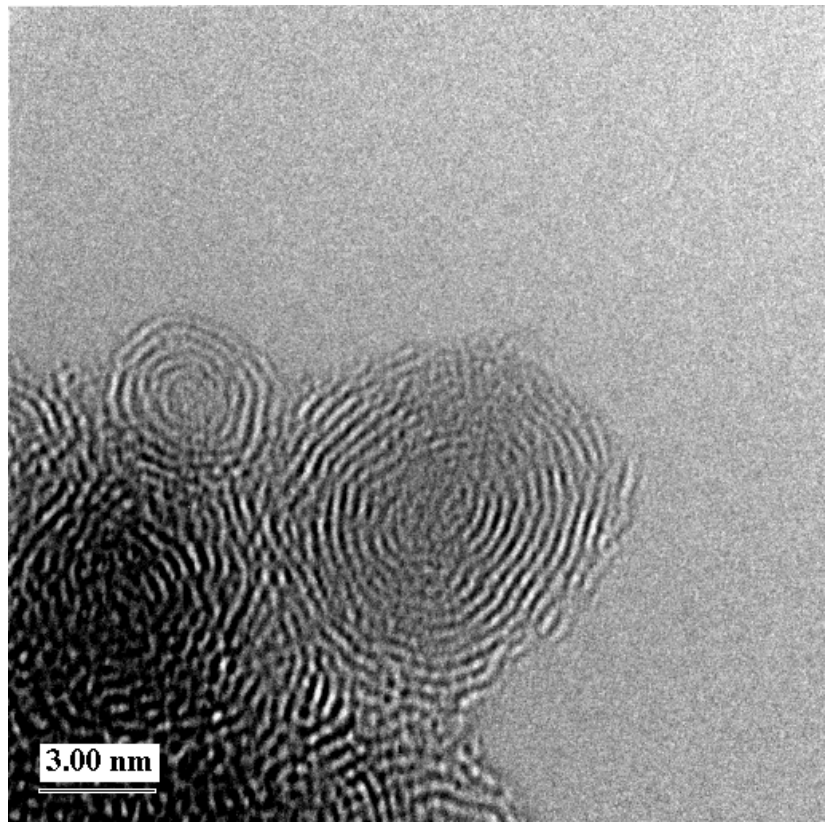
1 µm



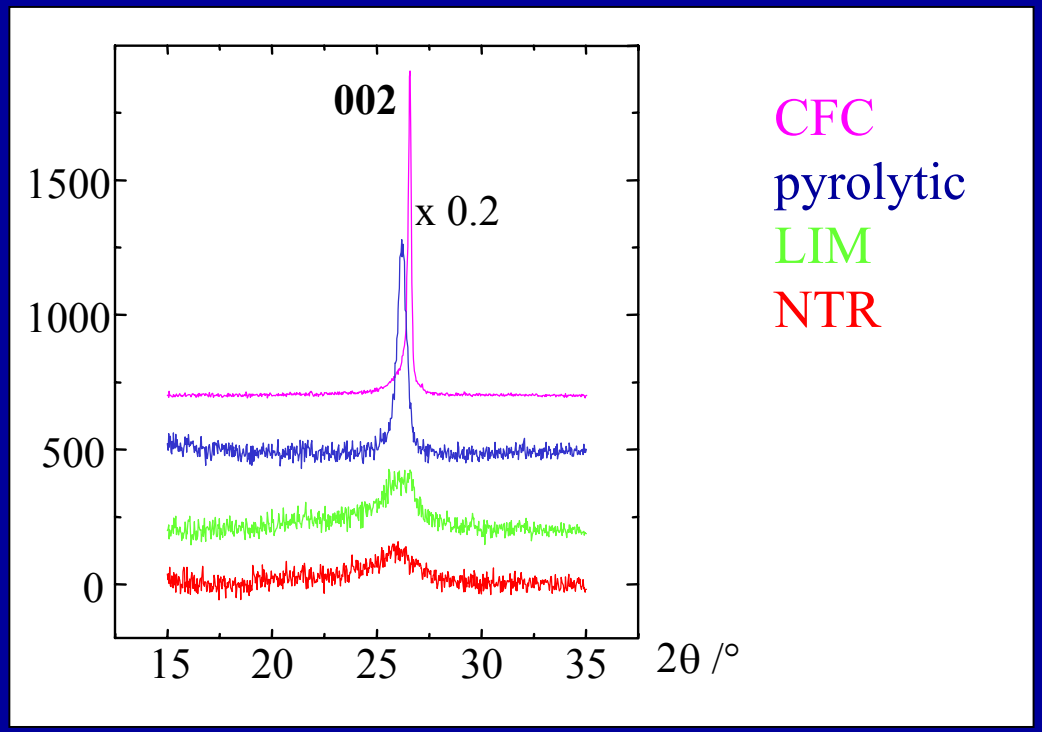
100 nm

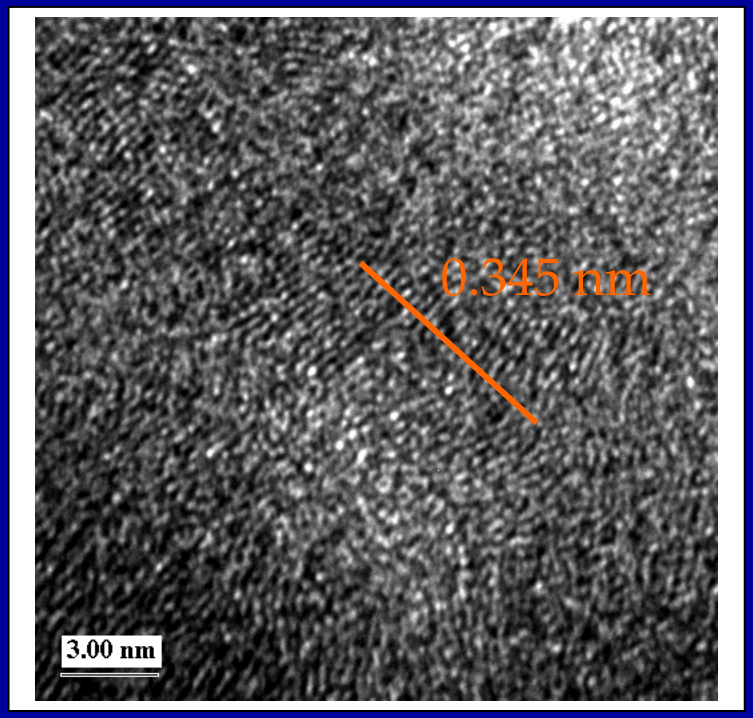
outboard limiter (VOL)





X-ray diffraction

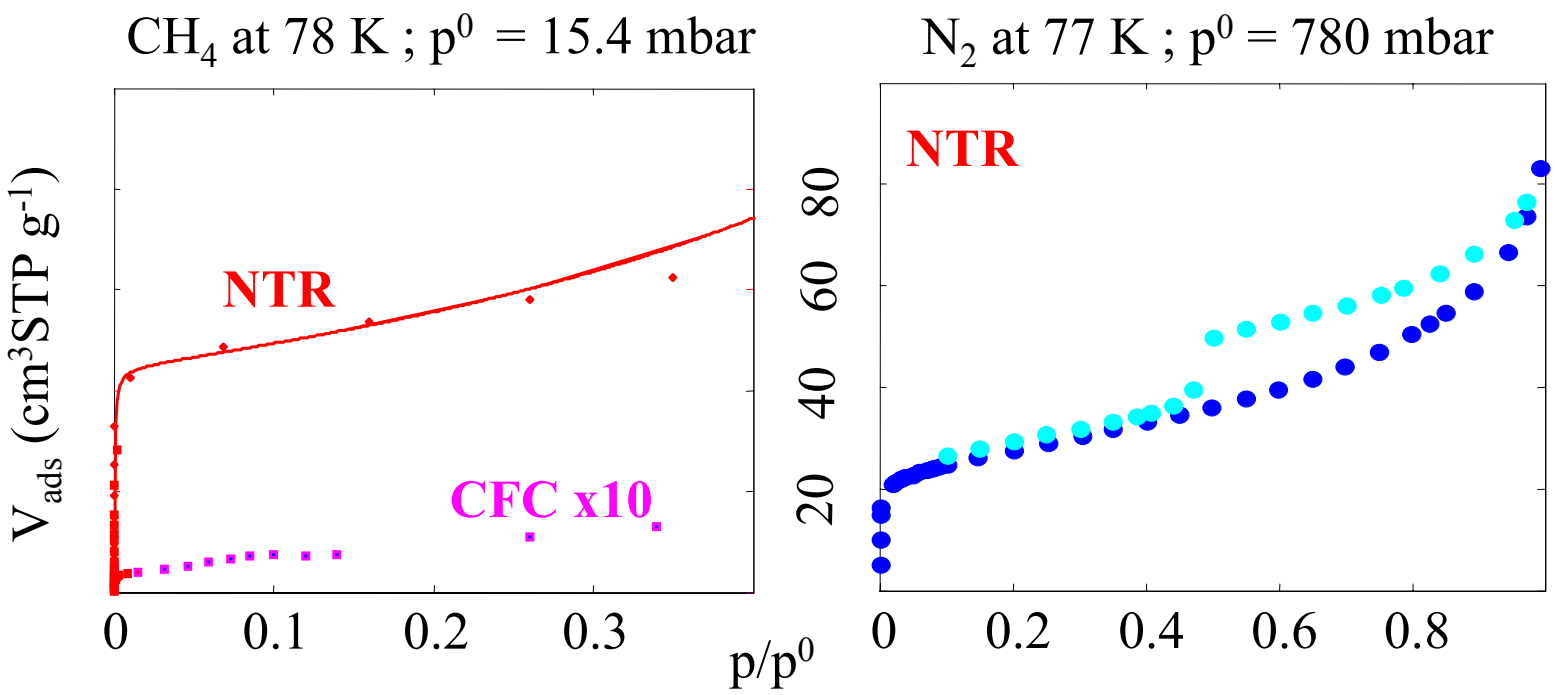




	2θ /°	d / Å	δ(2θ)	L_c / nm
CFC	26.54	3.36	0.23	74
pyrolytic	26.21	3.40	0.47	36
LIM	26.10	**3.41**	1.58	**11**
NTR	25.86	**3.45**	2.01	**8**

⇒ turbostratic graphite

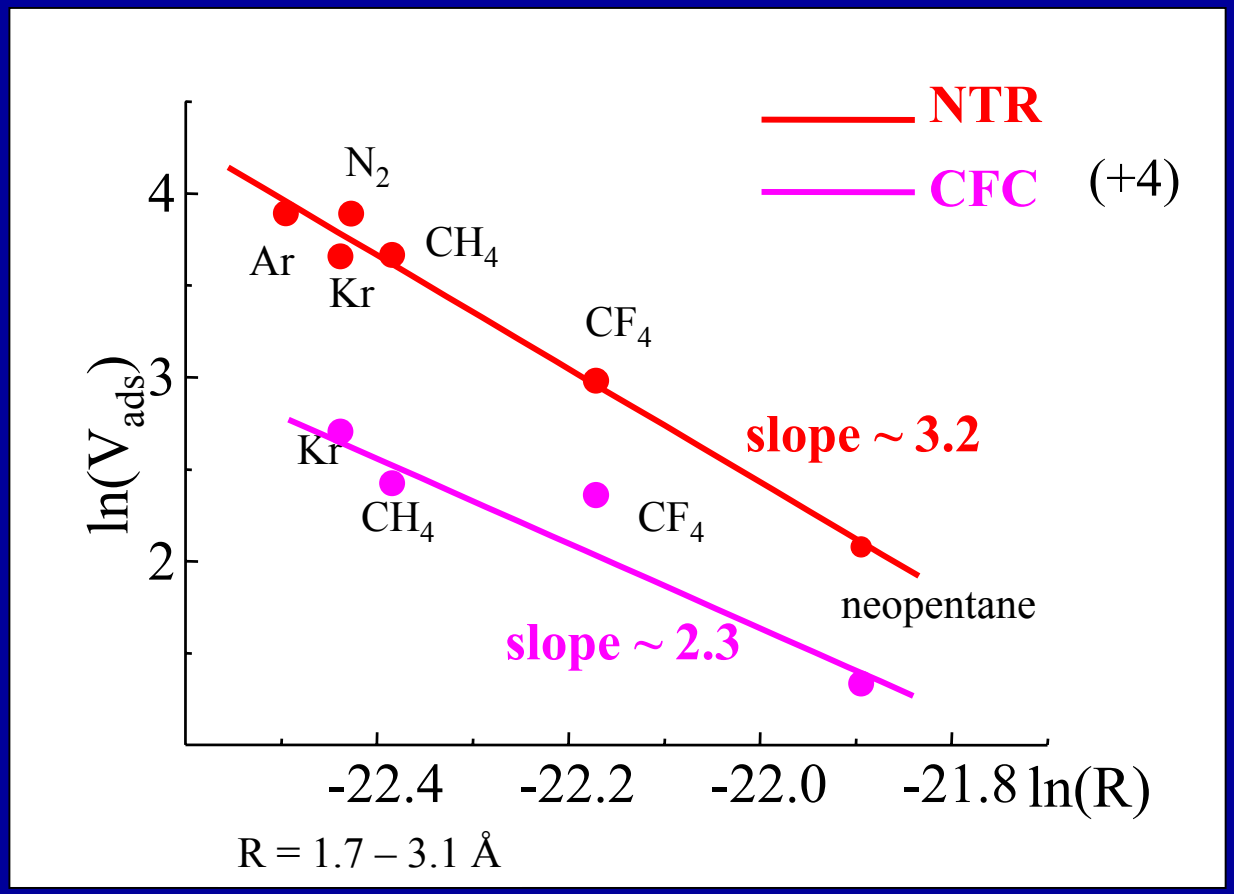
porosity analysis: adsorption isotherm volumetry



⇒ adsorption capacity much larger than CFC (x 30 - 100)
surface specific area 190 $m^2 g^{-1}$

⇒ evidence for slit-shape porosity (graphite-like)

"fractal" analysis

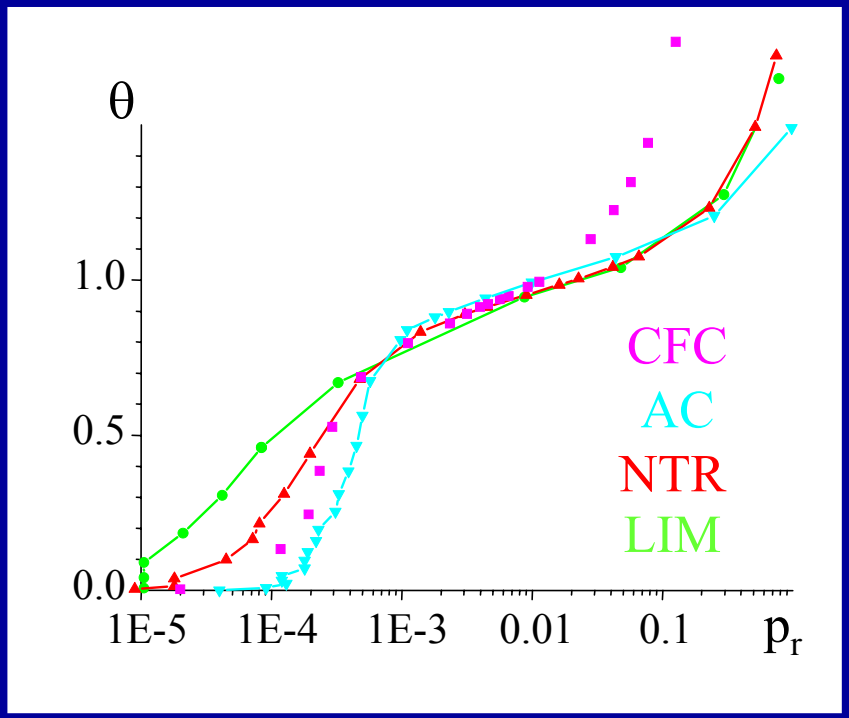


CFC: ~ surface
NTR: ~ volume

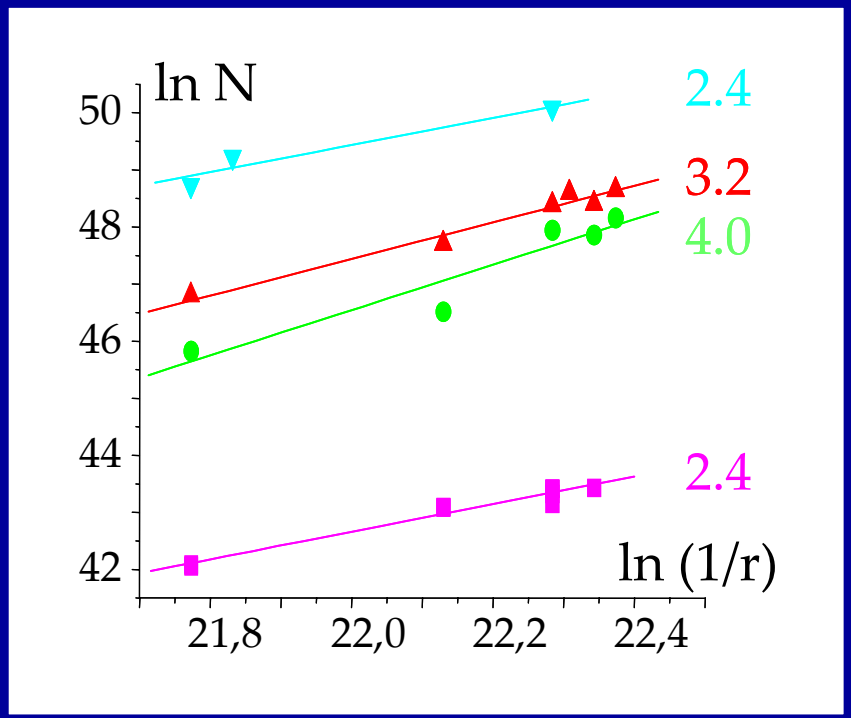
⇒ sub-nano scale: high disorder and pores of every size

comparison NTR / LIM

methane isotherms



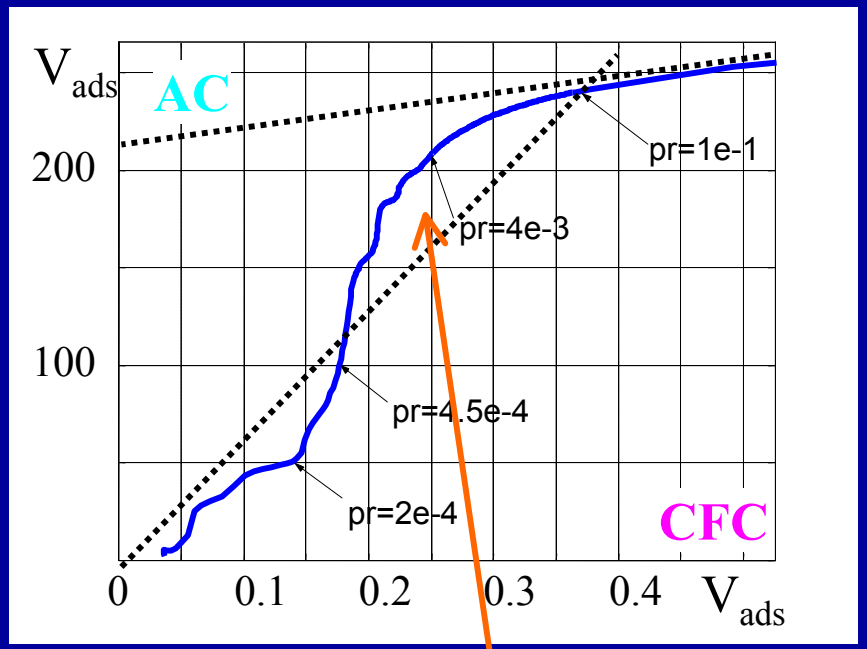
adsorbants of various sizes

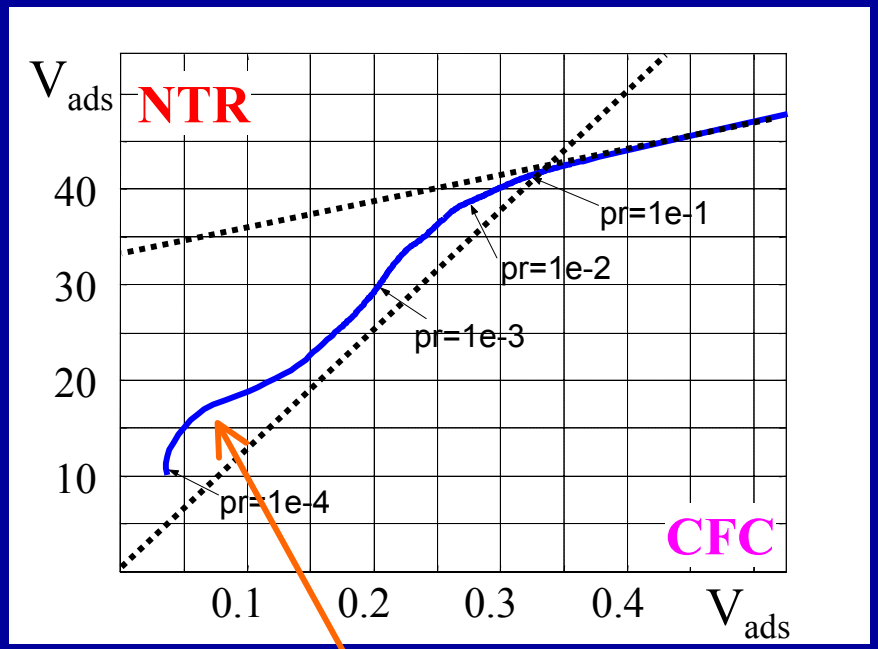


⇒ higher disorder for LIM than for NTR

α - plot

methane isotherms



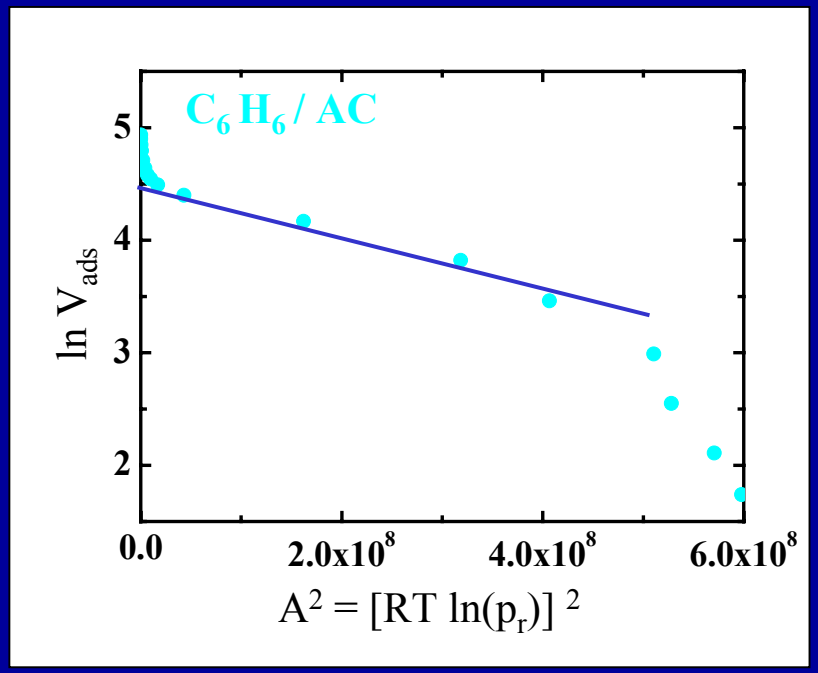


AC: super-micropores ≥ 1.4 nm

NTR: ultra-micropores ≤ 1 nm

pore-size distribution (Stoeckli method)

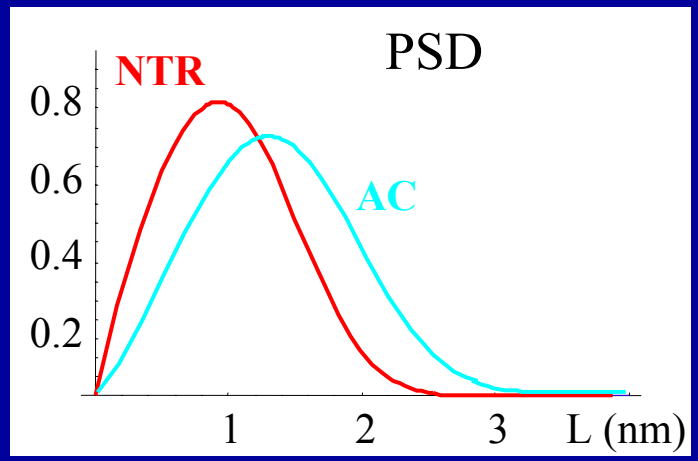
Dubinin: $V_{ads} / V_0 = \exp(-A^2 / \beta^2 E_0^2)$



Stoeckli: $V_{ads} / V_0 = [1 + (A/L_0E)^3/a]^{-m}$

PSD: dV/dL

$V_{ads} / V_0 = \int PSD\, dL$



linear fit:

C_6H_6: $E_0 = 19.9$ kJ mol^{-1} ; $L_0 = 1.3$ nm

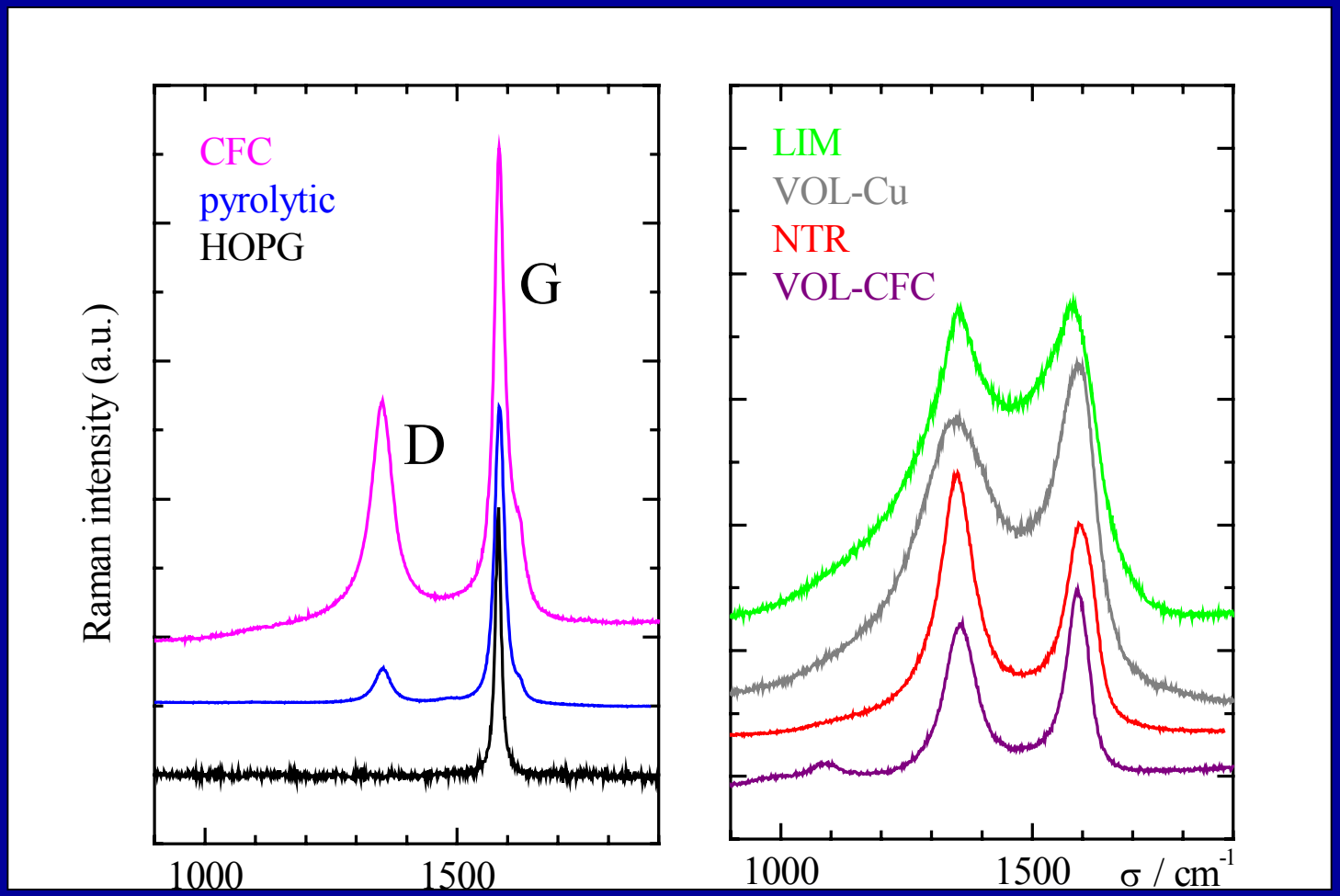
N_2: $E = 4.7$ kJ mol^{-1} ; $\beta = 0.24$

NTR: $E_0 = 23.7$ kJ mol^{-1} ; $L_0 = 0.9$ nm

AC: $L_m = 1.3 - 1.4$ nm

ntr: $L_m = 0.9$ nm

Raman microspectroscopy



- **G** Graphite-like band ↔ trivalent C atoms (sp^2)
- **D** Disordered-like band (sp^2) *or* mixed bonding sp^2 / sp^3

spectral decomposition

	G			D									
	P	w	I	P	w	I	P	w	I	P	w	I	I_D/I_G
HOPG	1582	14	100	-	-	0	-	-	0	-	-	0	-
pyrolytic	1584	22	82	1353	41	17	-	-	0	1625	8	1	0.2
CFC	1584	25	46	1351	52	45	-	-	0	1619	35	9	1.0
	1584	18	64	1353	52	30	-	-	0	1622	28	6	0.5
NTR	1598	**65**	24	1350	87	**55**	**1524**	124	16	1226	186	**5**	2.3
VOL	1597	**70**	13	1347	196	**58**	**1545**	157	25	1145	253	**5**	4.5
VOL	1592	**52**	32	1357	74	**57**	**1534**	78	12	-	-	**0**	1.8
LIM	1595	**88**	16	1350	110	**32**	**1505**	179	23	1244	414	**29**	2.1
LIM	**1586**	**105**	17	1354	130	**25**	**1487**	105	14	1248	472	**44**	1.5
LIM	**1576**	**123**	24	1354	143	**29**	**1478**	150	26	1238	312	**21**	1.2

⇒ G and D bands: signature of a non crystalline graphite-like carbon

⇒ G and D widths: highly disordered carbon ($L_{//}$ = 1 - 3 nm)

⇒ additional band at 1500 cm^{-1}: more sp^3 defects for LIM than NTR

main results and perpectives

deposit properties

low D / C ratio (RMN, NRA, FTIR)

10 % for cold deposits

1 % for hot deposits

local graphite-like structure (XANES, Raman, X-ray diffraction, TEM)

highly disordered $\leftrightarrow$ D^+ bombardment

open porosity: surface: 200 $m^2\ g^{-1}$

volume: 0.05 $cm^3\ g^{-1}$

volume: 10 %

$\Rightarrow$ dynamics

input parameters for a multiscale analysis of the diffusion of H, H_2 inside the wall

- financial support: Euratom-CEA association

- collaborations

  - Ph Parent, C. Laffon (LURE - Orsay) XANES
  - Ph Colomban, G. Sagon (LADIR - Thiais) Raman
  - F. Ziarelli, S. Caldarelli (TRACES - Marseille) NMR
  - W. Saikaly, CP2M -Marseille TEM
  - service commun St Charles - Marseille SEM
  - J.-P. Astier CRMCN - Marseille X-ray diff.