Outgassing studies of stainless steel and materials for a detector system in the CESIUS storage ring

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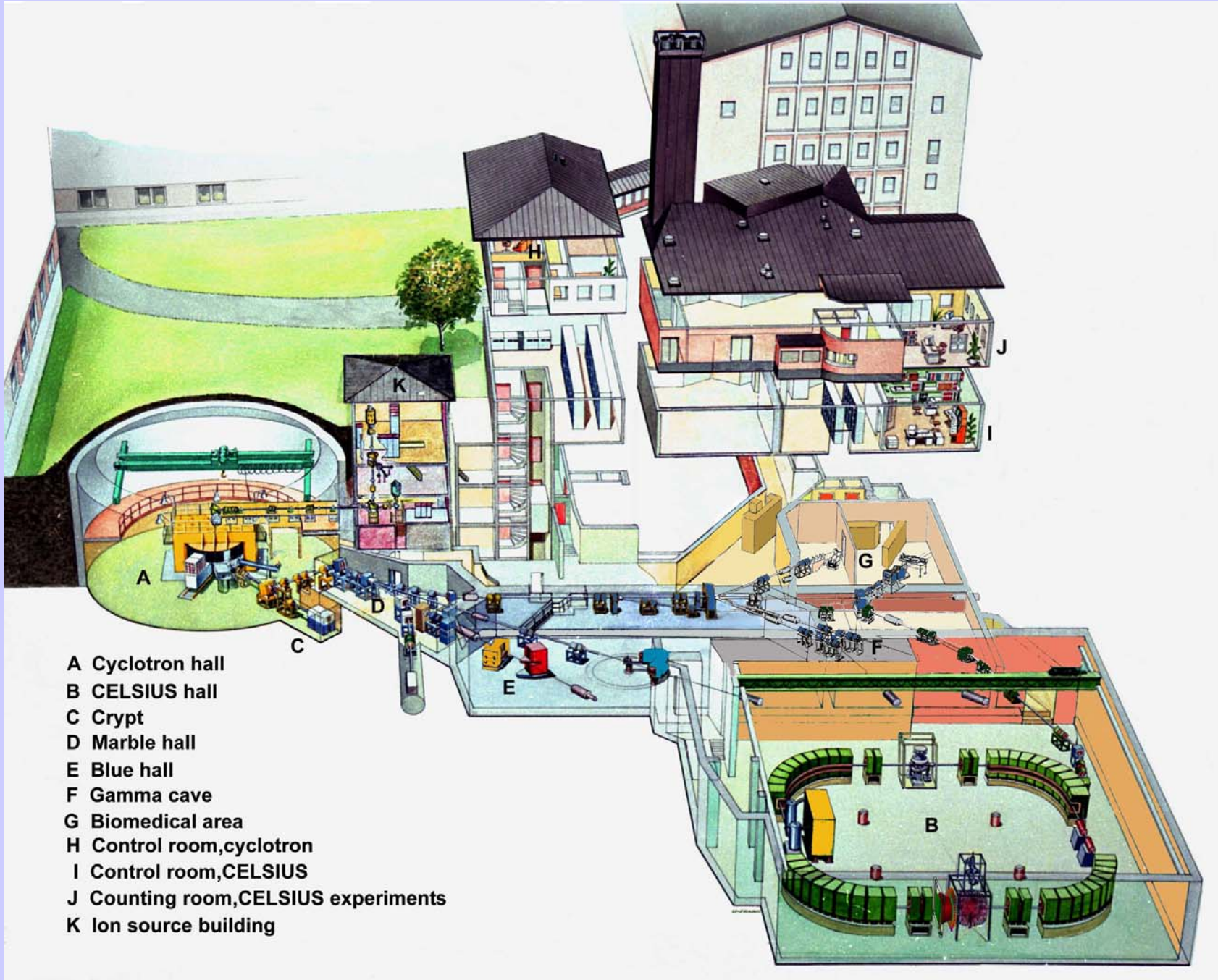
Layout

The Svedberg Laboratory

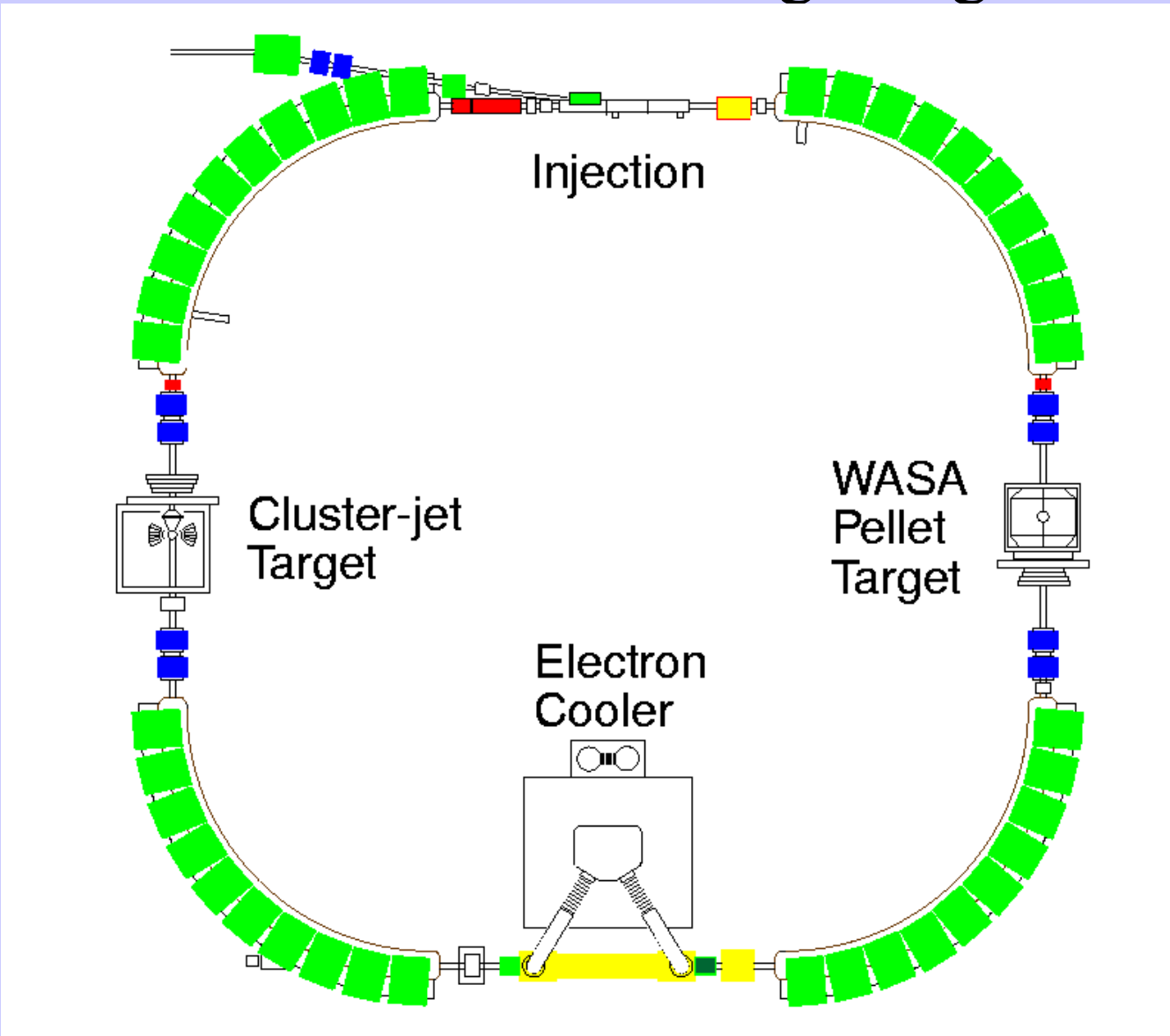
The CHICSi detector system

UHV materials development for CHICSi

Outgassing of stainless steel and hydrogen bulk concentrations


A Cyclotron hall
B CELSIUS hall
C Crypt
D Marble hall
E Blue hall
F Gamma cave
G Biomedical area
H Control room,cyclotron
I Control room,CELSIUS
J Counting room,CELSIUS experiments
K Ion source building

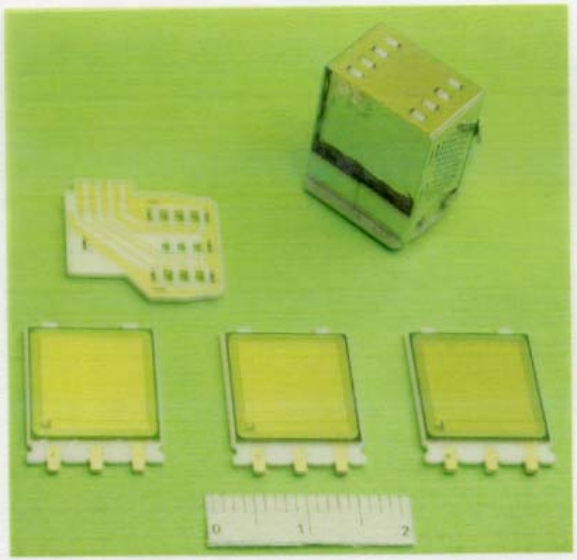
CELSIUS storage ring



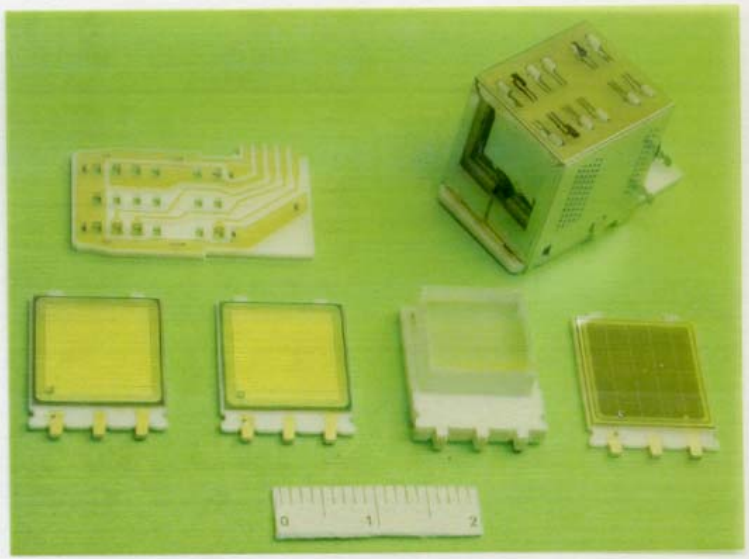
CHICSi - a new UHV-compatible system of silicon and time-of-flight particle detectors for heavy-ion reaction experiments at a storage ring

- *540 detector modules, each with 3-4 elements, Silicon diodes or scintillators*
- *Identify particles from heavy-ion reactions*
- *Low-energy particles → No wall or intermediate vacuum possible*
- *VLSI readout chip in UHV*

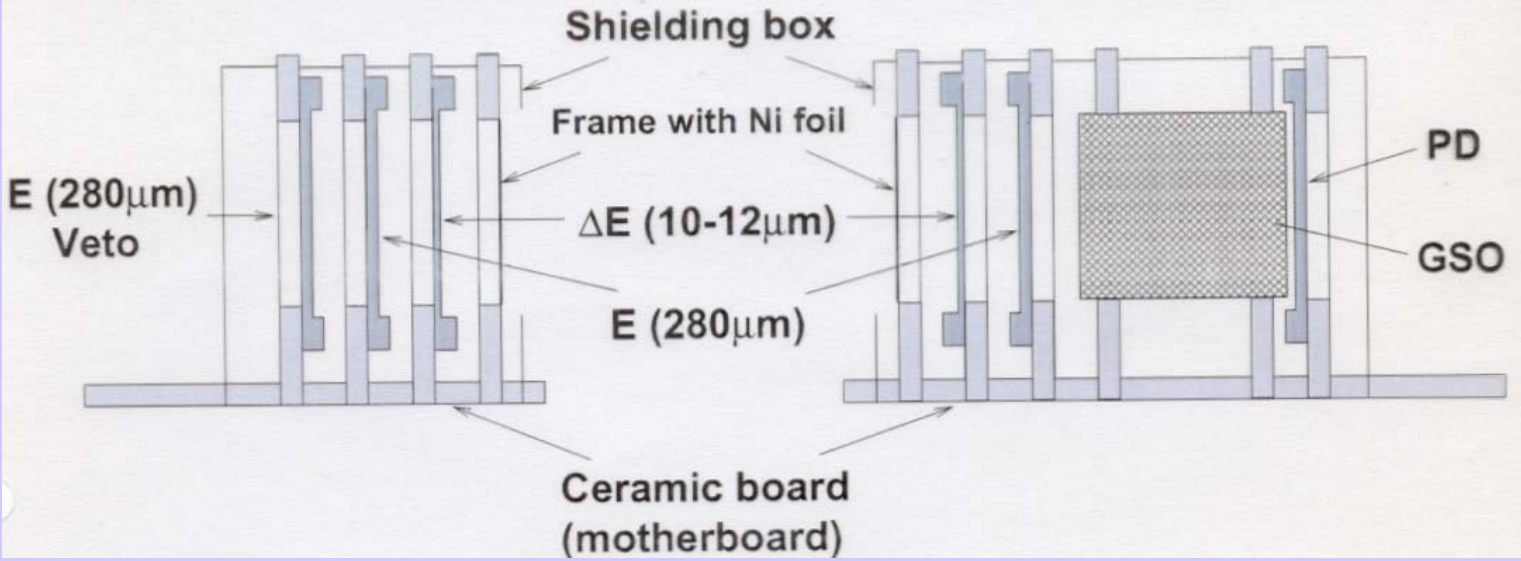
Particle-detector telescopes

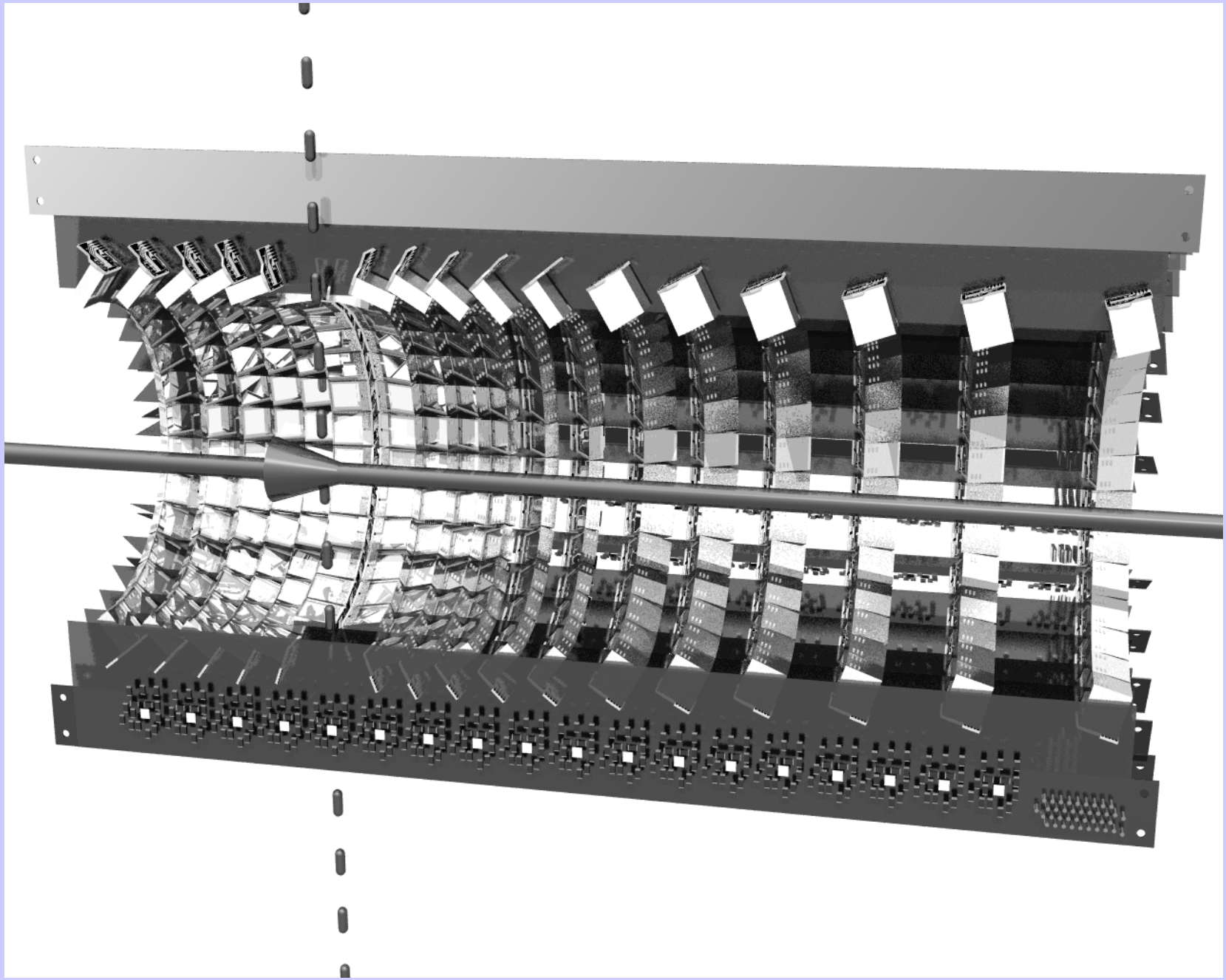


Large Angle Telescope

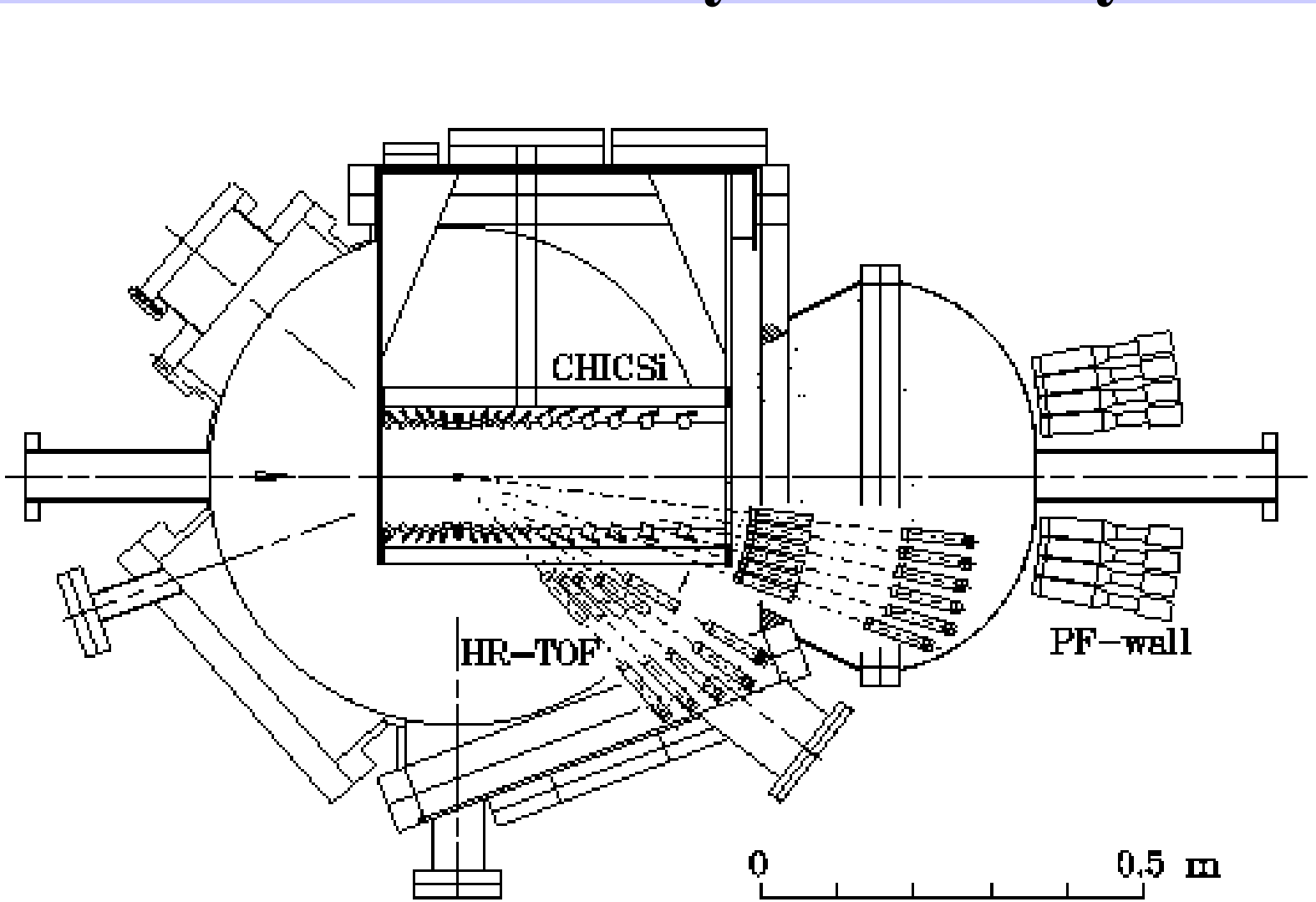


Forward Angle Telescope





CHICSi and auxiliary detector systems



UHV compatible materials needed

- *New type of rectangular flanges for large ports*
- *Printed circuit boards for*
  - *detector mounts*
  - *VLSI readout*
- *Coaxial cables*
- *Flat cables*
- *Contacts for cables and printed circuit boards*
- *Conducting and insulating two-component glues*

Outgassing measurements

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A. Wegner

The Svedberg Laboratory and
Department of Radiation Sciences
Uppsala University

and CHICSi development group

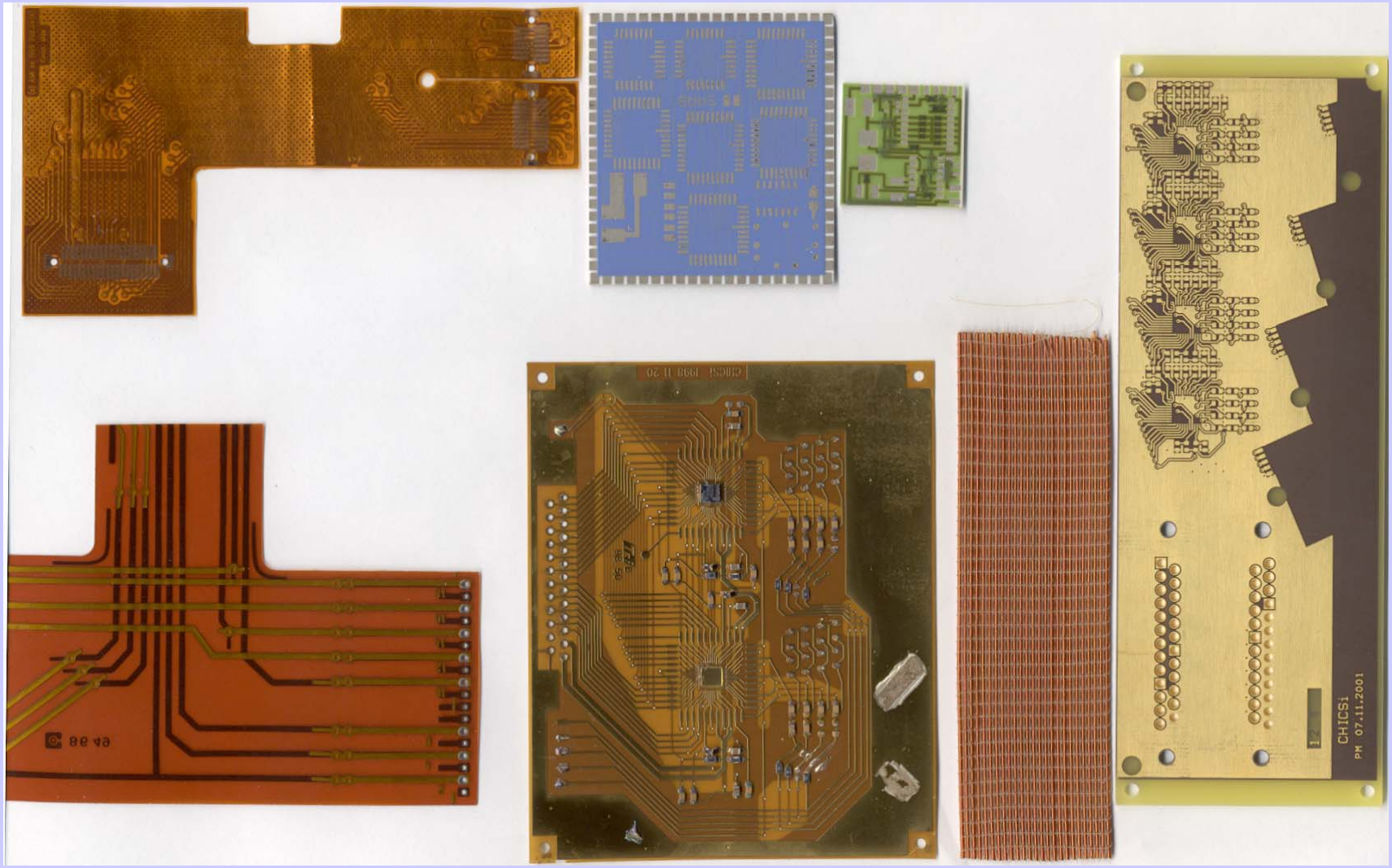
Insulators

- Macor
- PEEK
- Vespel
- Photoveel
- M-soft shapal

Insulator materials

Macor	Can break when machining Threads are fragile
Vespel	Varying outgassing Discharge can cause carbonizing
PEEK	High outgassing, H_2O
Photoveel	SiO2, Al_2O_3 ... Lower outgassing
M-soft Shapal	AlN Low outgassing, Expensive

Kapton Ceramics FR-4



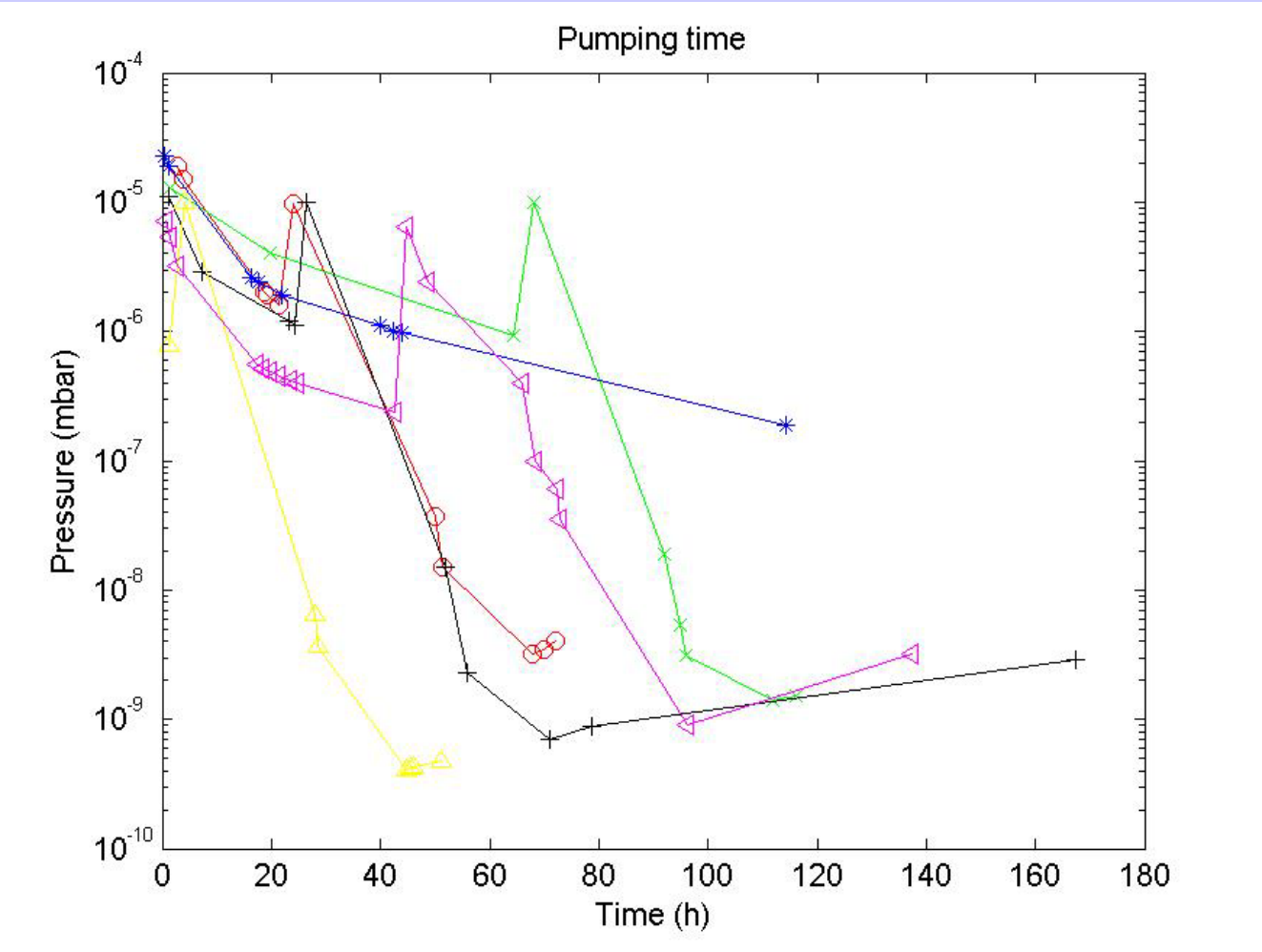
Outgassing measurements

Material	Area	Bake-out in air		Bake-out in vacuum		H_2	H_2O	CO	Total outgassing
	cm^2	Temp °C	Time h	Temp °C	Time h	W/m^2	W/m^2	W/m^2	rate W/m^2
PRINTED CIRCUIT BOARD MATERIALS									
Pyralux (2 layers)	19			150	20	$2 \cdot 10^{-5}$	$4 \cdot 10^{-7}$	$3 \cdot 10^{-6}$	$3 \cdot 10^{-5}$
AP (2 layers)	98			150	20	$2 \cdot 10^{-7}$	$4 \cdot 10^{-8}$	$3 \cdot 10^{-7}$	$1 \cdot 10^{-6}$
Epoxy-Acrylic (2 layers)	30			150	20	$1 \cdot 10^{-7}$	$3 \cdot 10^{-7}$	$3 \cdot 10^{-7}$	$3 \cdot 10^{-6}$
Epoxy-Glass fibre, 1 layer	36			150	20	$4 \cdot 10^{-7}$	$4 \cdot 10^{-8}$	$4 \cdot 10^{-8}$	$2 \cdot 10^{-6}$
Alumina (14 layer electrical print)	32			150	20				$3 \cdot 10^{-7}$
Glass reinforced Kapton™	74			150	24				$2 \cdot 10^{-8}$
FR4	475			150	30				$1.5 \cdot 10^{-8}$
FR4	331	150	24						$6 \cdot 10^{-7}$
FR4	331	150	24	150	23	$2 \cdot 10^{-10}$			$6 \cdot 10^{-8}$
FR4	331	150	24	150	77				$4 \cdot 10^{-9}$
FR4 – 6 layers	120			150	30	$<1 \cdot 10^{-7}$			$5 \cdot 10^{-7}$
INSULATORS									
PEEK				150	20	$1.4 \cdot 10^{-6}$	$8 \cdot 10^{-7}$	$5 \cdot 10^{-7}$	$3 \cdot 10^{-6}$
Photoveel	44			150	20	$1.6 \cdot 10^{-7}$	$1.0 \cdot 10^{-8}$	$3 \cdot 10^{-8}$	$2 \cdot 10^{-7}$
M-soft shapal	44			150	20	$1.4 \cdot 10^{-8}$	$1.0 \cdot 10^{-9}$	$4 \cdot 10^{-9}$	$1 \cdot 10^{-7}$
Macor™	75			200	26				$4 \cdot 10^{-8}$
Macor™, 30 min air expos	75								$2 \cdot 10^{-6}$

Outgassing measurements Epoxies (EPO-TEK)

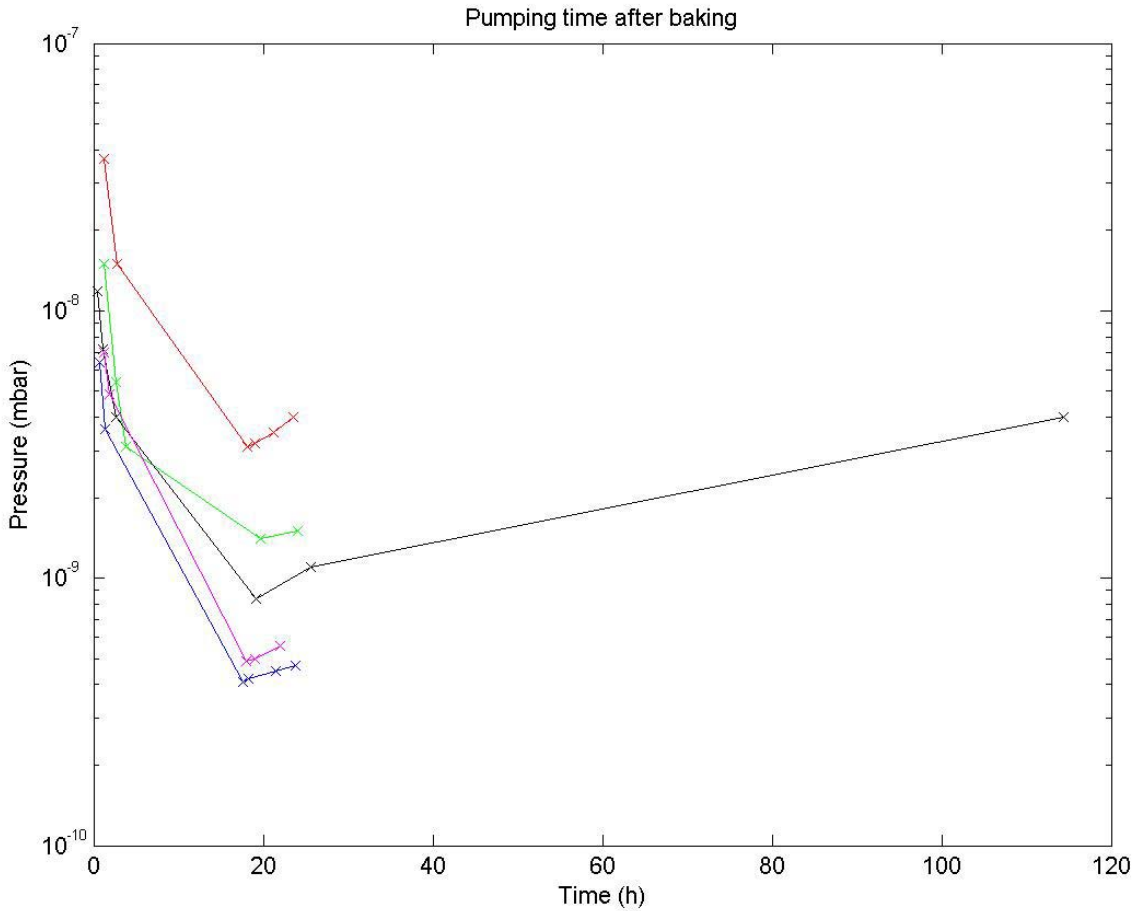
Type	Weight	Bake-out in air		Bake-out in vacuum		H_2	H_2O	CO	CO_2	CH_4	Total outgassing rate
	(g)	Temp (°C)	Time (h)	Temp (°C)	Time (h)	(W/g)	(W/g)	(W/g)	(W/g)	(W/g)	(W/g)
377	1.15	90	1.5								$1.4 \cdot 10^{-7}$
377	1.15	90	1.5	150	24	$1.3 \cdot 10^{-10}$	$1.8 \cdot 10^{-11}$	$2.0 \cdot 10^{-11}$	$4 \cdot 10^{-12}$	$6 \cdot 10^{-12}$	$4 \cdot 10^{-10}$
H20E	2.16	90	1.5			$4 \cdot 10^{-8}$	$1.6 \cdot 10^{-8}$	$6 \cdot 10^{-9}$	$8 \cdot 10^{-10}$		$6 \cdot 10^{-8}$
H20E	2.16	90	1.5	150	24	$4 \cdot 10^{-11}$	$1.2 \cdot 10^{-10}$	$3 \cdot 10^{-11}$	$2 \cdot 10^{-11}$	$1.4 \cdot 10^{-11}$	$7 \cdot 10^{-10}$
H27D	3.90	90	1.5								$1.1 \cdot 10^{-7}$
H27D	3.90	90	1.5	150	24						$3 \cdot 10^{-8}$

Outgassing from cables



Red o AMGAB cable
Green x KAP2
Blue * KAP2 airbaked inst. after 48h delay
Black + KAP3
Yellow ^ Empty
Magenta< KAP2 airbaked inst. in chamber directly

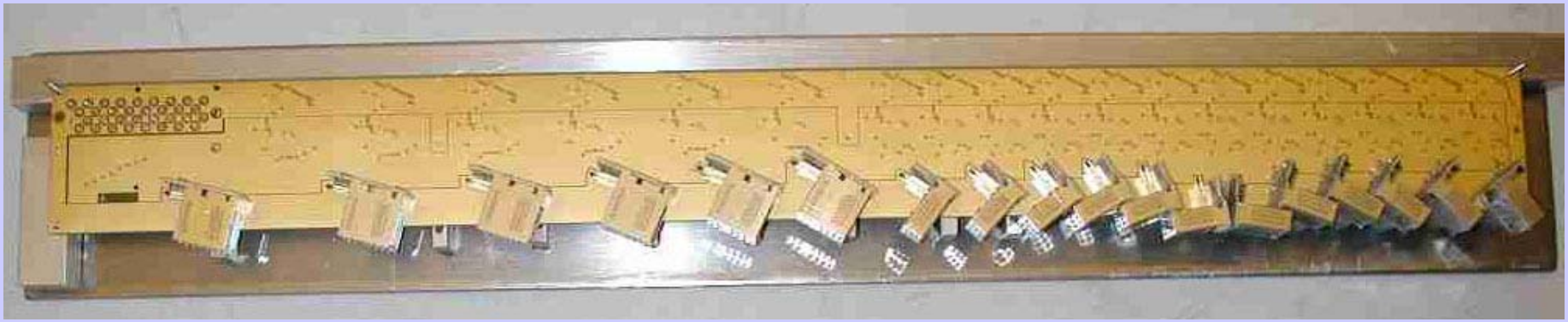
Outgassing from cables

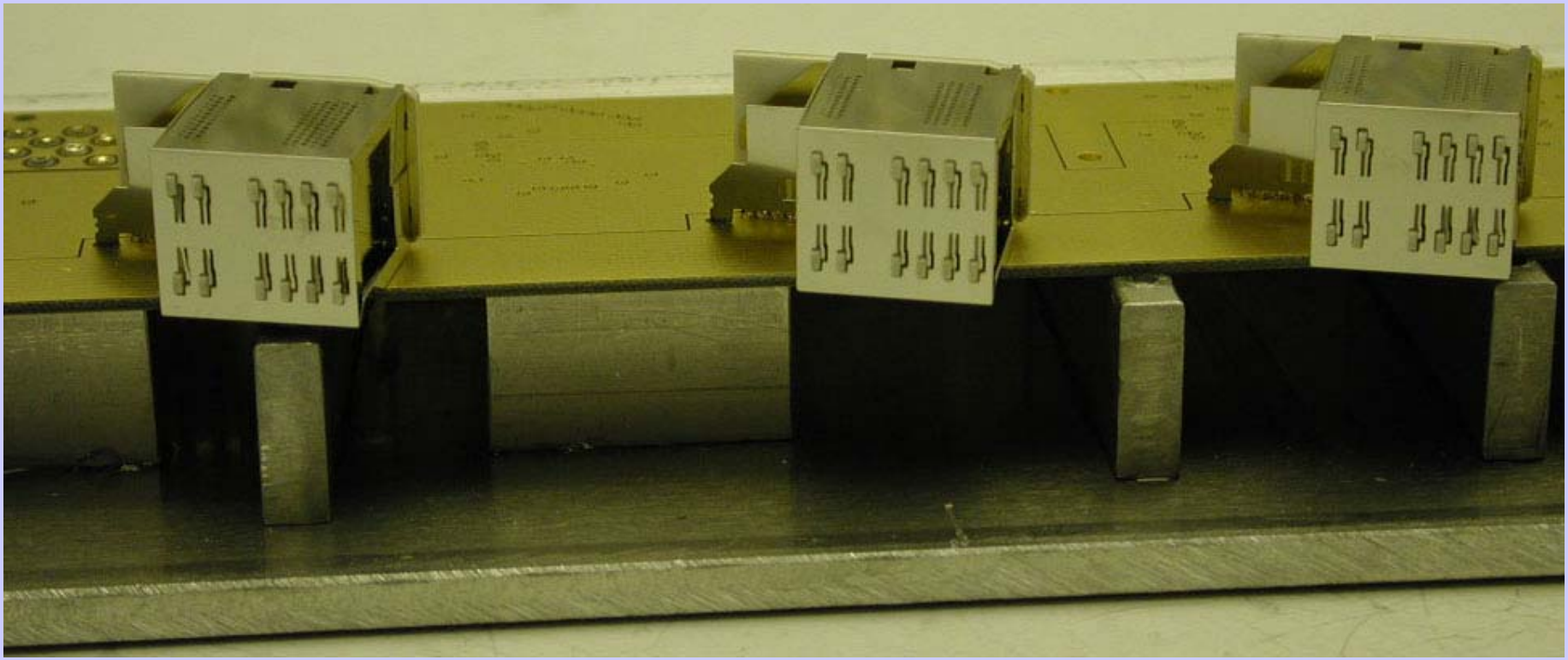


Red o AMGAB cable
Green x KAP2
Blue * Empty
Black + KAP3
Magenta< KAP2 RGA changed

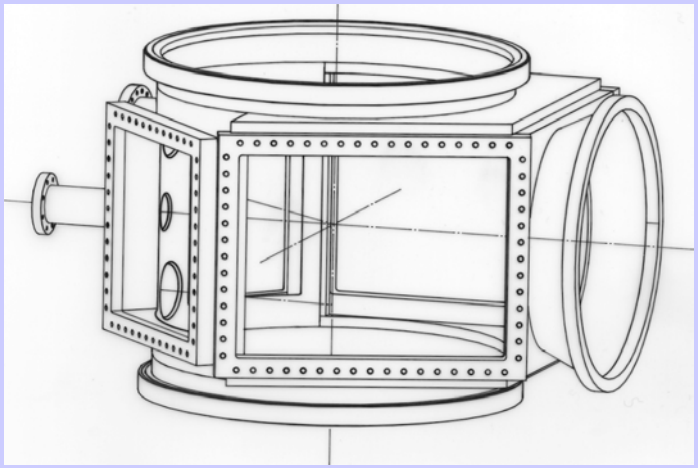
Time after finished baking

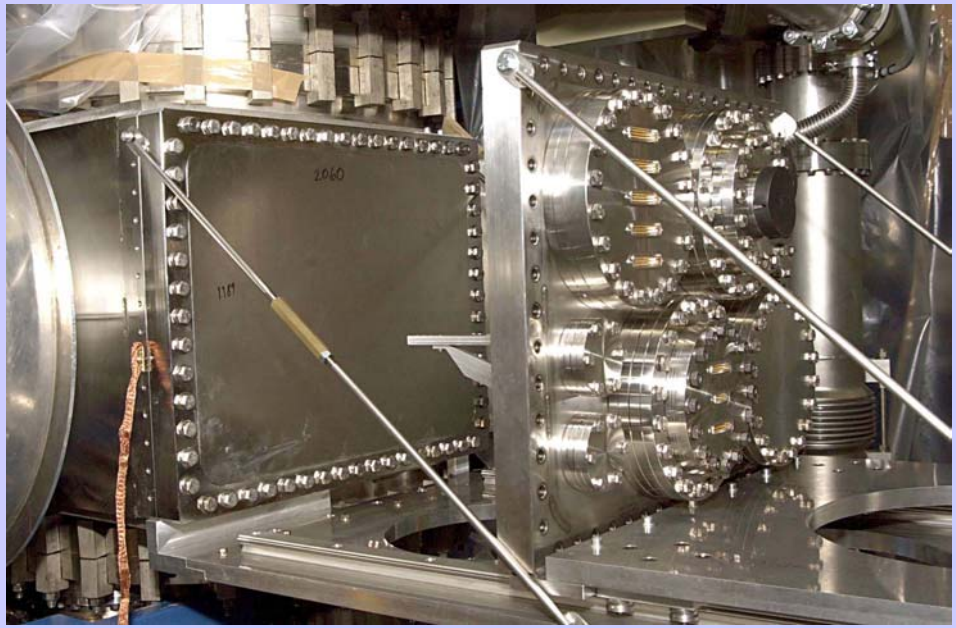
GrandMother board





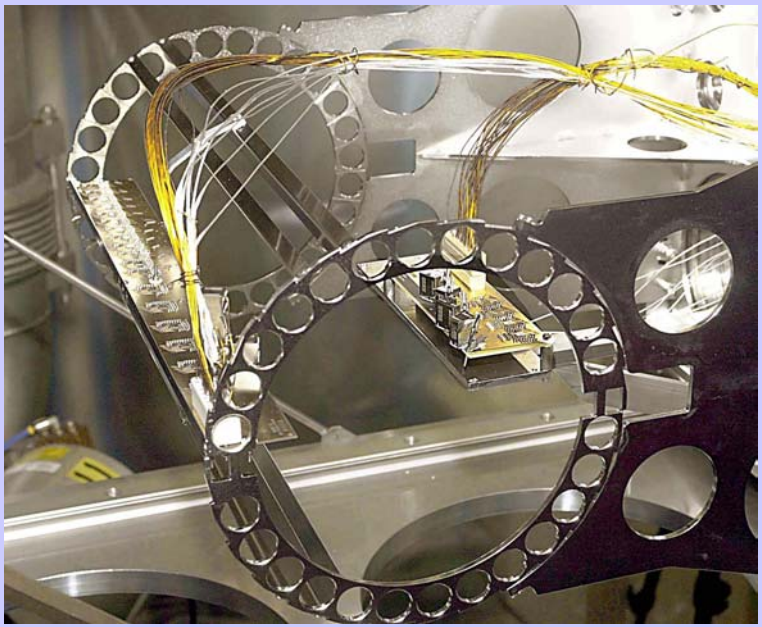
Scattering chamber



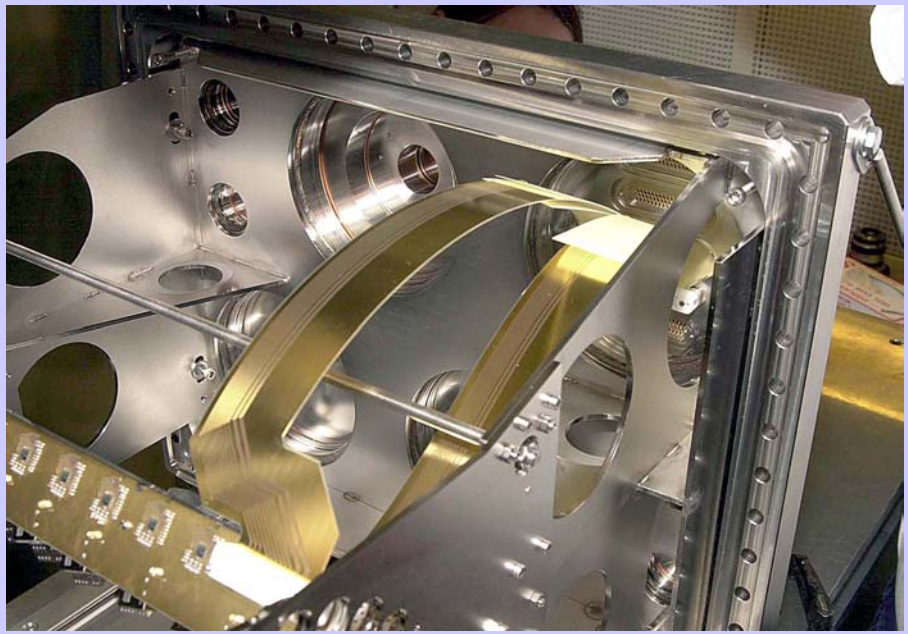


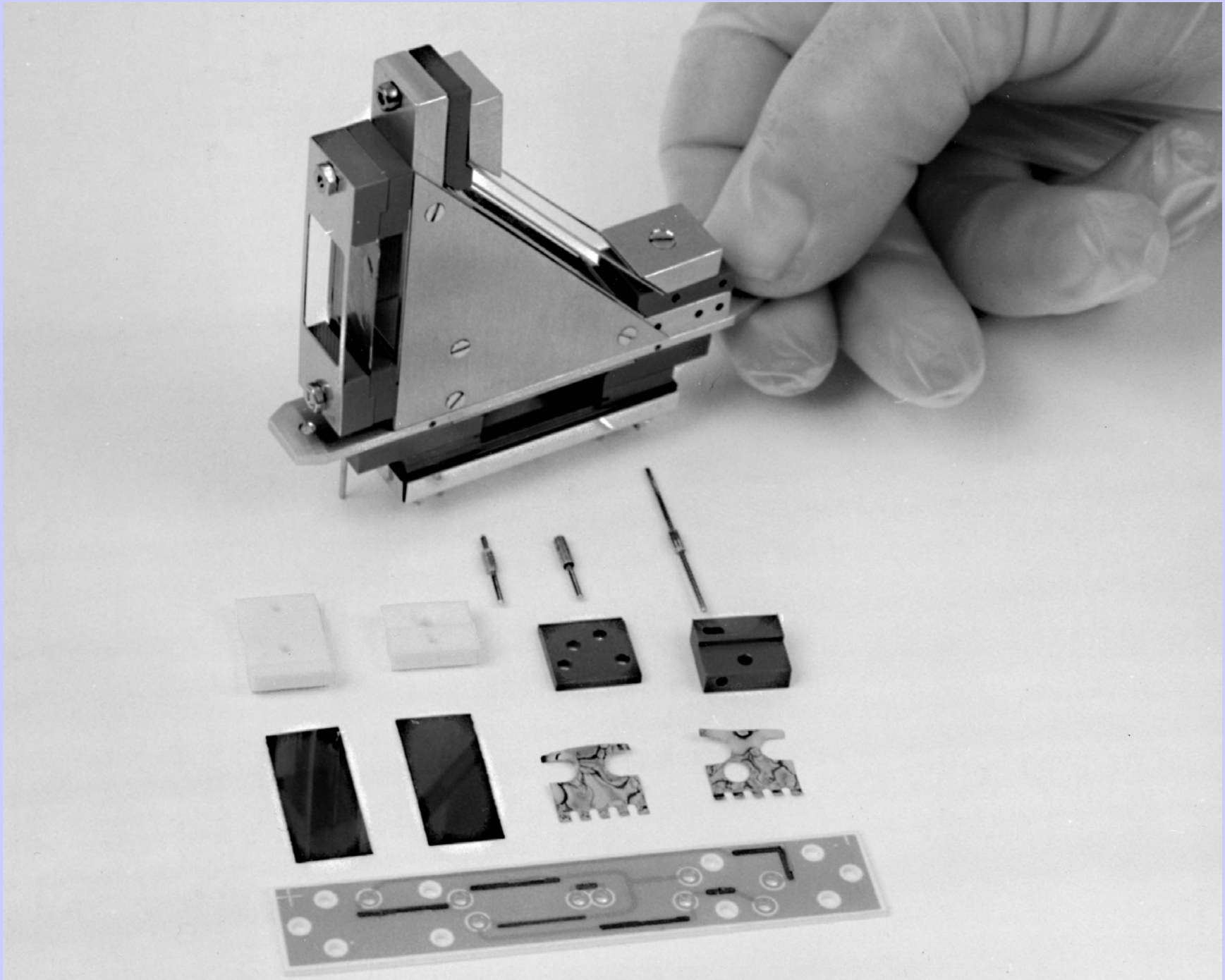
Readout cables

Kapton-insulated cables



Printed-circuit board cable





Conclusions

- New outgassing data on insulators, glue, printed circuit boards, cables.
- Flexible printed-circuit board cables are not yet reliable
- It is important to perform outgassing measurements before building complex systems

Hydrogen profile experiments

Tandem accelerator 6 MV

^{15}N beam

Resonance reaction $^{1}H(^{15}N, ^{4}He\ \gamma=4.3\ MeV)^{12}C$

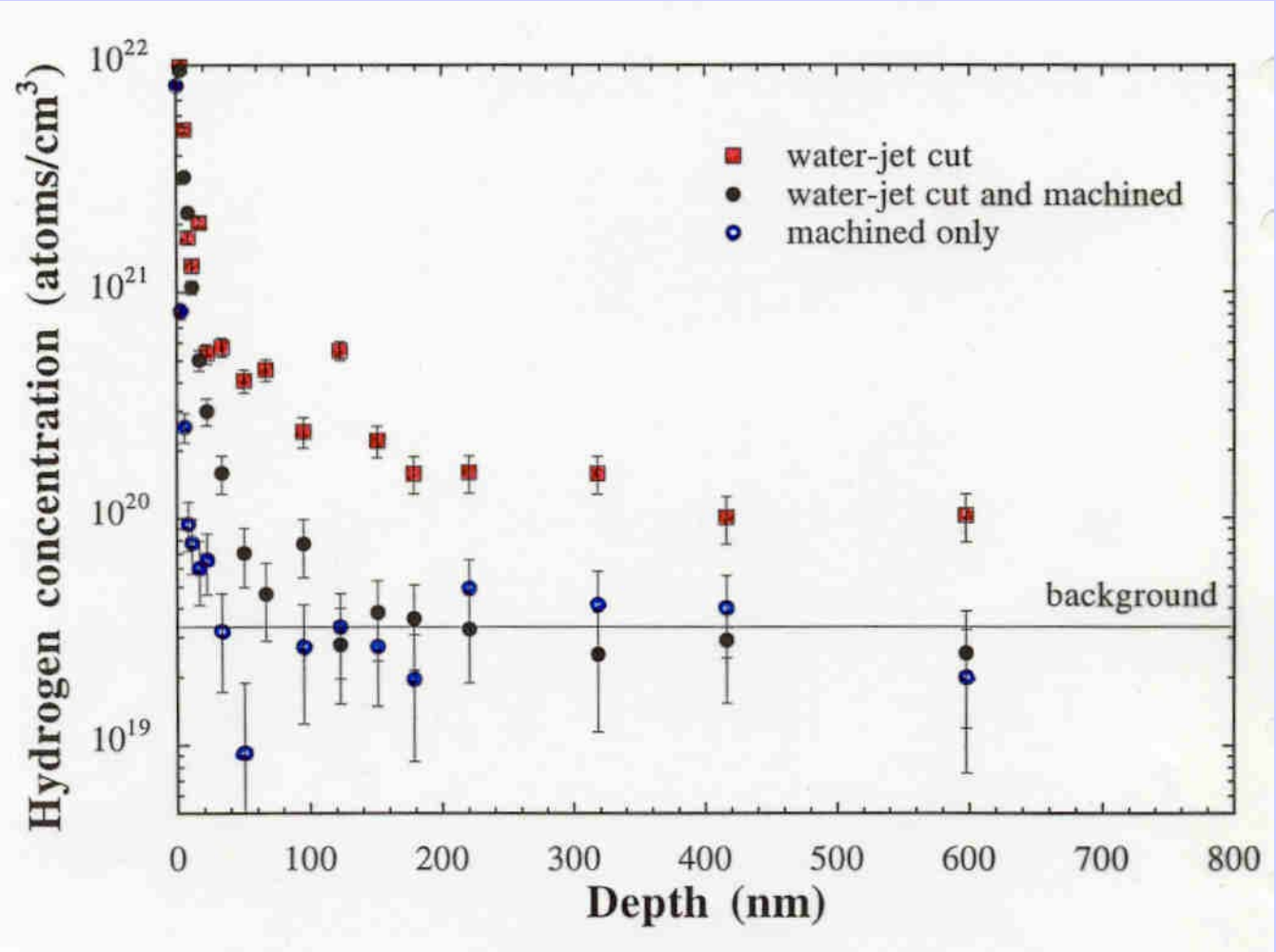
Method:

W A Lanford et al. Appl Phys Lett 28(1976)566

Calibration

L Westerberg et al Nucl Instr and Meth B9(1985)49

Hydrogen profiles



L. Westerberg and B. Hjörvarsson Vacuum 47(1996)687

Studies of hydrogen profiles in stainless steel

L. Westerberg, B. Hjörvarsson
Uppsala University
E. Wallén and Mathewson,
CERN

Vacuum 48(1997)771

Sample preparation and extraction experiments

24 samples 1cm^2, 2mm, 304L low carbon X3 Cr Ni 18.10

Standard CERN cleaning procedure

Air bake in furnace at 400 °C 2, 4, 8, 16 and 24 h

Vacuum firing at 950 °C 10^{-4} – 10^{-3} Pa 1h,
cooled fast 300 °C/h

Extraction measurements at Fischer & Rosemount GmbH, Hanau, Germany
Samples heated to 1000 °C, kept there for 3 min
A flow of N_2 brings desorbed H_2 to a counting unit
(3 min is enough acc. to F&R to bring out all H_2)

Results

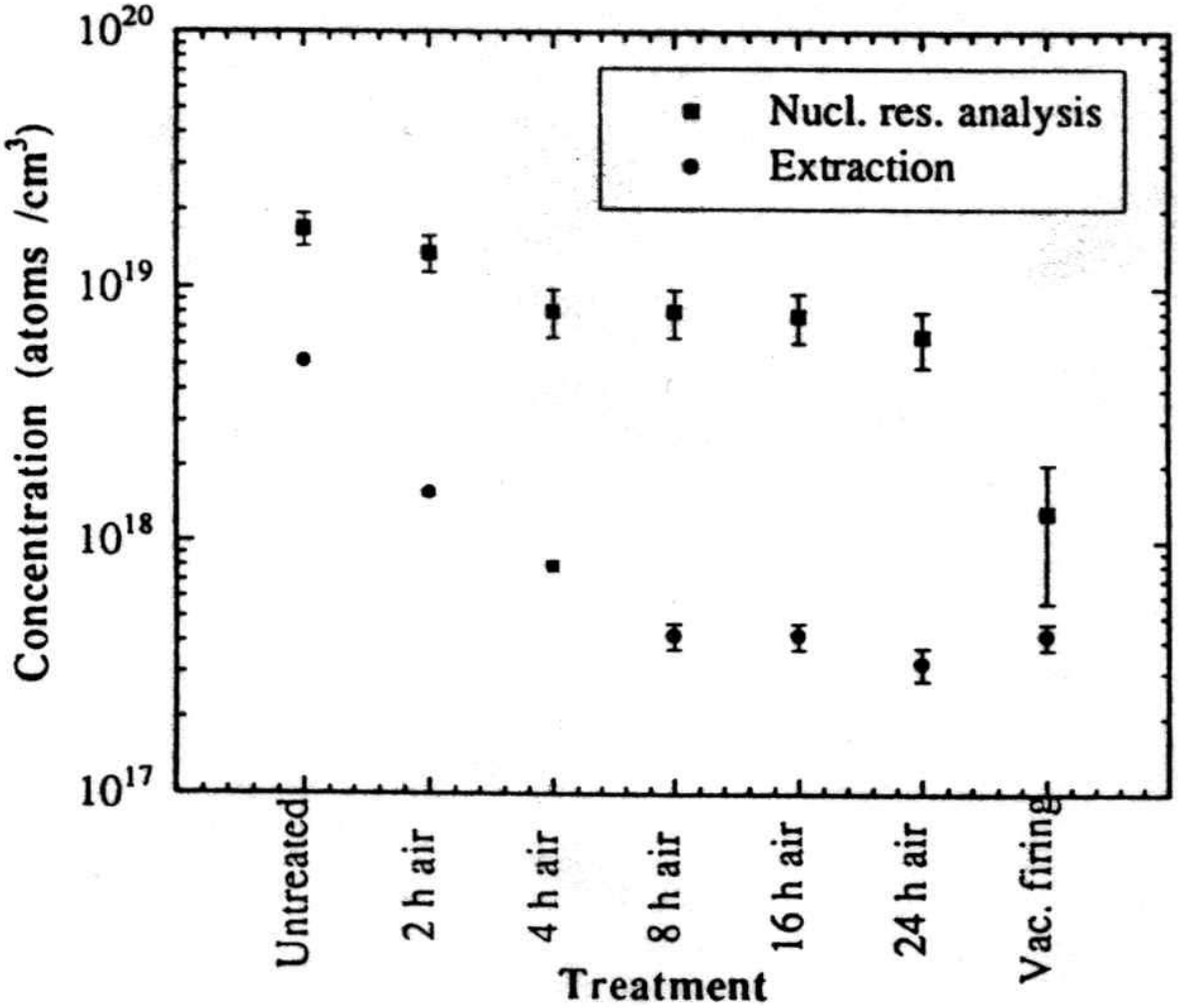


Figure 1. The average hydrogen concentration in the 0.05–0.7 μm depth range of the virgin, air baked and the vacuum fired samples. The results from the extraction method are also included.

Results & Interpretation

Air bake: > 4 h same H level.

Reduction by a factor of 2.

Vacuum fired: Reduced a factor of 10.

Extraction: Lower H

Interpretation

- Not all H is extracted
- Origin of reduced outgassing assigned to deep trapping of hydrogen in the bulk and/or formation of diffusion barriers – not to the reduction of the hydrogen content in the material

Outgassing and Hydrogen Content of Untreated or Vacuum Fired Stainless Steel after Bakeout or Airbake to 300-450 °C

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N. Hilleret, B. Versolatto and J. P. Bojon
CERN, Switzerland
B. Hjörvarsson
Uppsala, Sweden

Experimental procedure
In situ bake out to 300 ºC

is carried out in the following way to take into account the large thermal inertia of the stack of stainless steel sheets:

The temperature is ramped up to 300 ºC during 24 hours. Then it is kept constant for 24 hours

The temperature is then decreased down to 150ºC for the outgassing of the analyzer. The origin of the time is taken when the temperature is lower than 30 ºC.

Experimental procedure

Air bake at 450ºC

The system temperature is raised to 450ºC during 24 hours, the system being at atmospheric pressure and filled with laboratory air. After the 24 hours period, the system is evacuated and baked to 300ºC according to the above procedure.

Vacuum Firing

carried out at 950ºC during
2 hours at a H_2 partial pressure
lower than 10^{-3} Pa

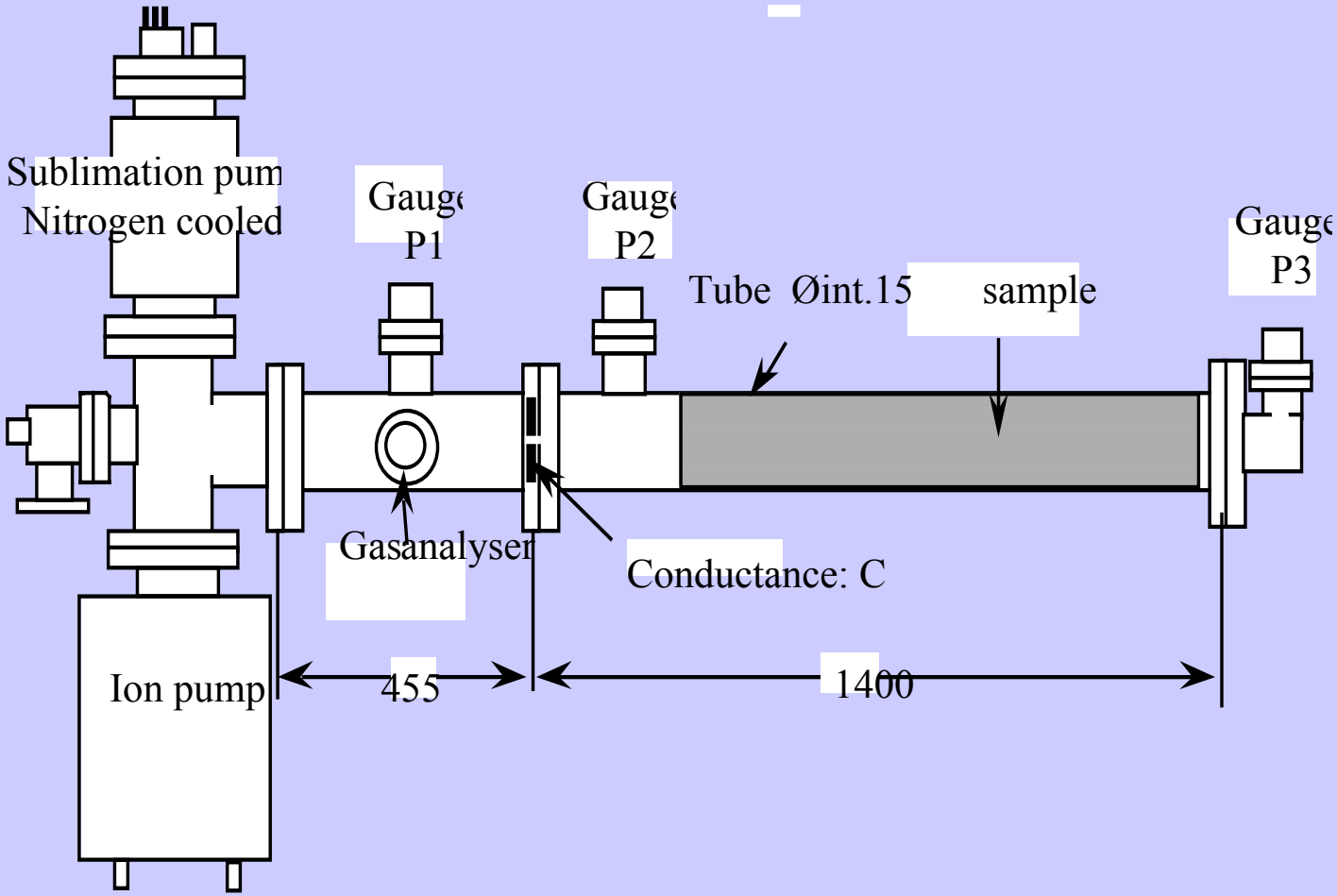
Experimental procedure

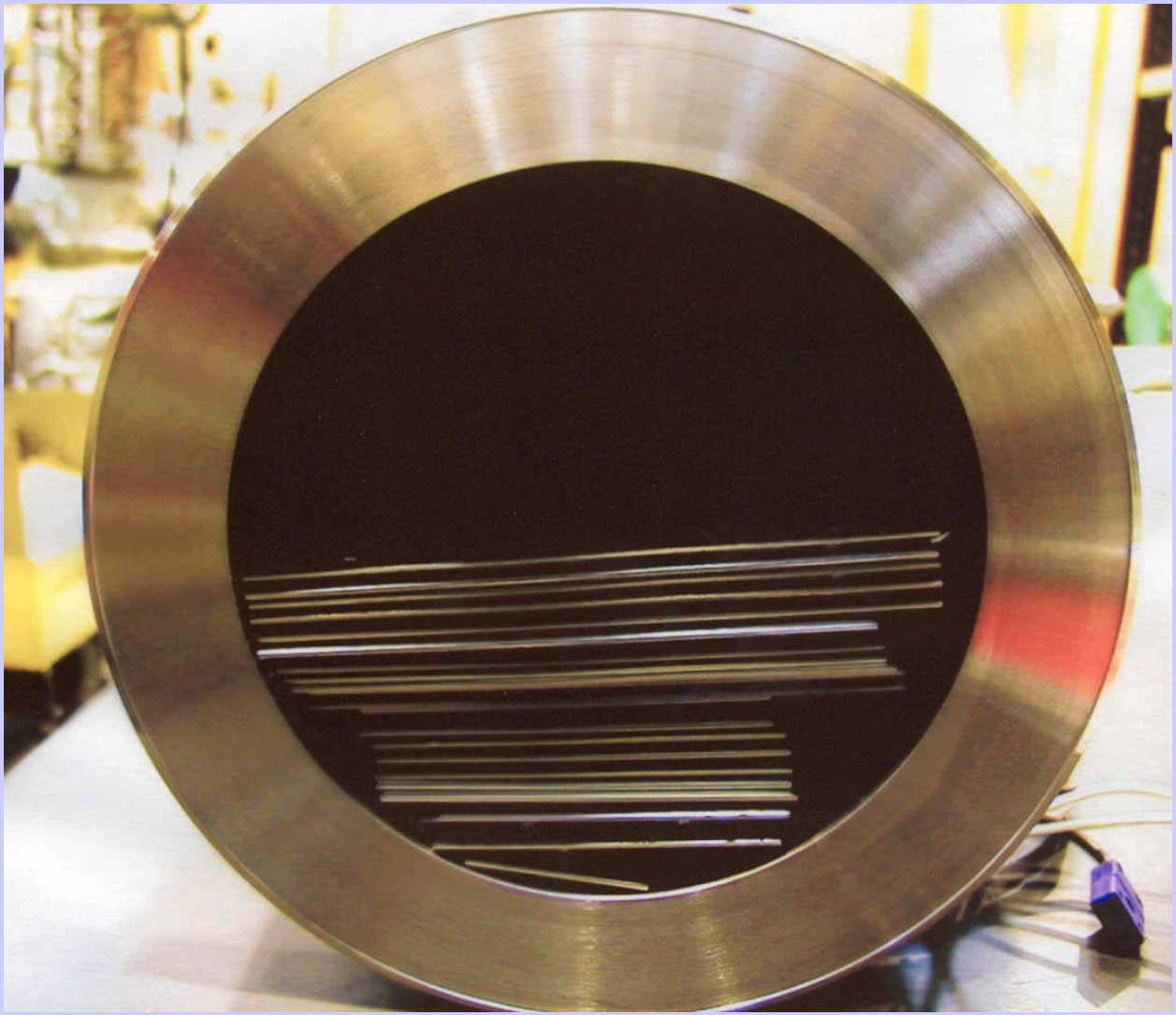
SAMPLES

The samples where stacks of stainless steel sheets (1.5 mm thick, total area 13.5 m^2 , each separated by a 1.6 mm clearance) which have been degreased in perchlorethylene vapour, immersed in an alkaline detergent bath rinsed in demineralised water and dried in a furnace.

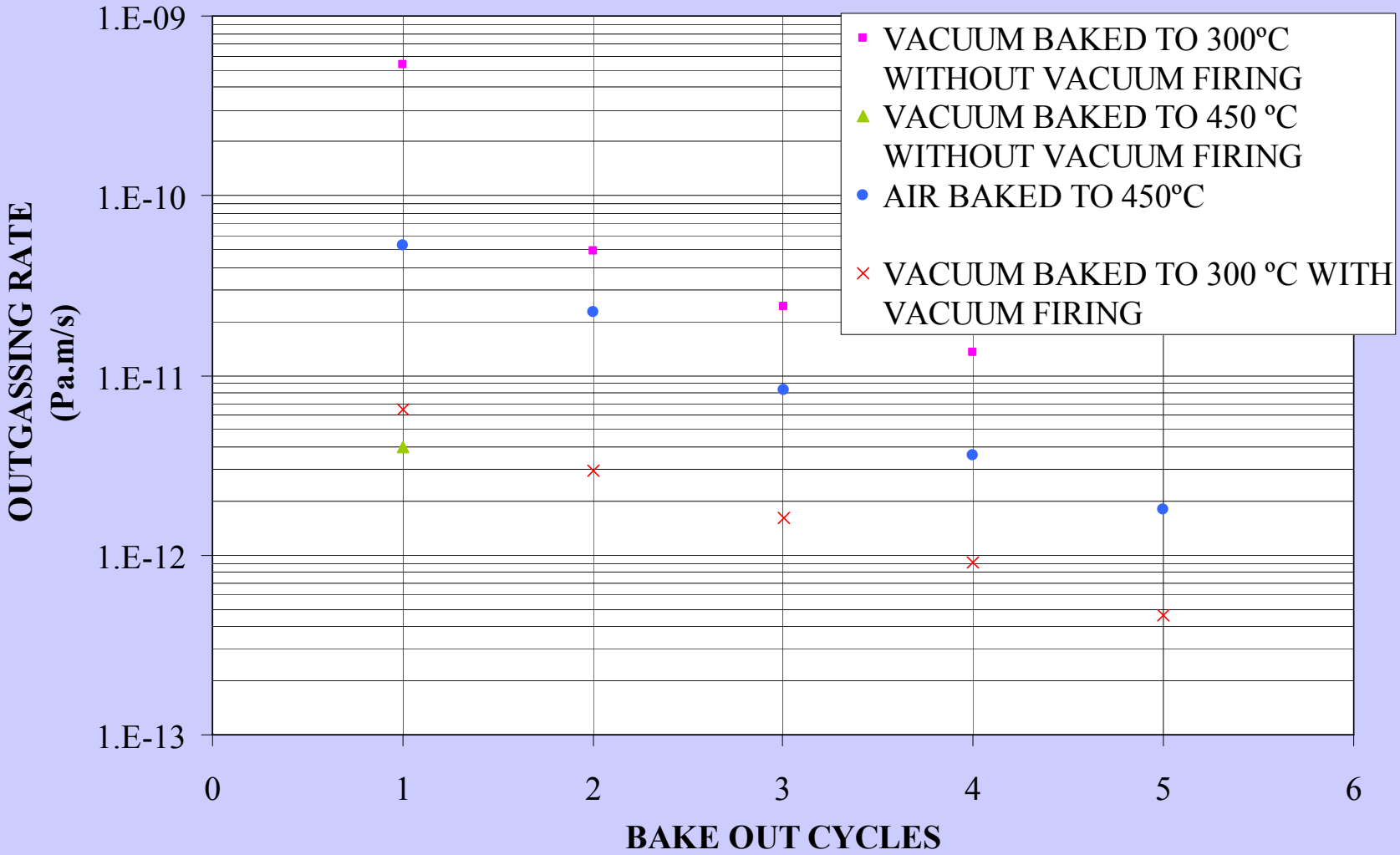
The ratio a/A where a is the pumping speed of the system expressed in an equivalent area with a sticking coefficient unity and A is the total outgassing area (13.5 m^2) is 0.113

THE EXPERIMENTAL SET UI





HYDROGEN OUTGASSING RATE AFTER VARIOUS TREATMENTS (MEASURED 120 HOURS AFTER BAKE OUT)



Some conclusions from a talk by Paulo Chiggiato, CERN, given at the AVS congress in Denver 2002.

- TDS measurements of sst 316LN, 316L, 304L
  Different H2, H2O, CH4 and CO2
- H2 charging when electroplating after vacuum firing
- Reduced outgassing after vac firing of H2, H2O, CH4 and CO2
- Lower outgassing when vacuum firring in 10-7
- Vacuum firing does not reduce e- stimulated desorption

(Priv. comm, not yet publ.)

Further plans

Study hydrogen conc. of these samples with

- hydrogen profile method
- and neutron beam analysis

European network on vacuum science

- Appl for research training network
- 10 partners