Investigation of materials developed and used at CERN by microscopy.

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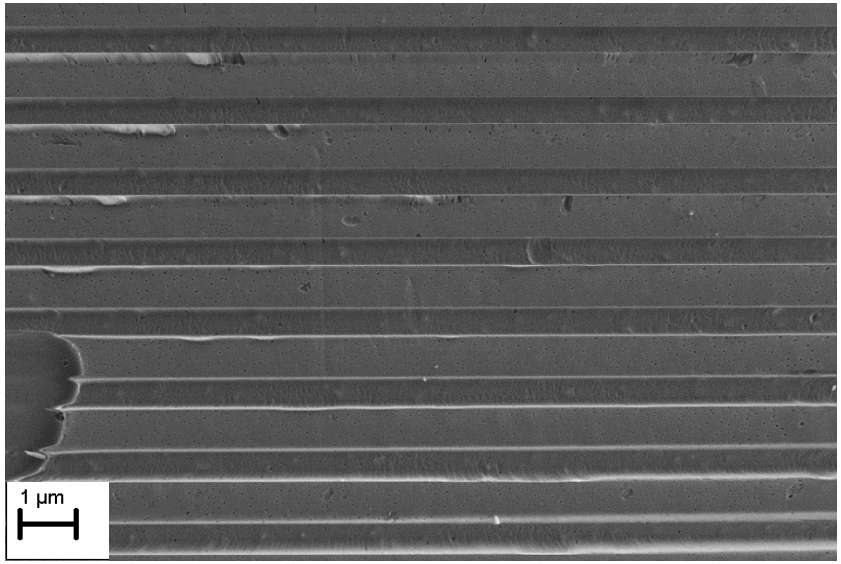
WAMAS, CERN 20. November 2013

Outline:

1. SEM introduction
2. Typical SEM
3. EDS and EBSD
4. Materials analysed at CERN

1. SEM introduction

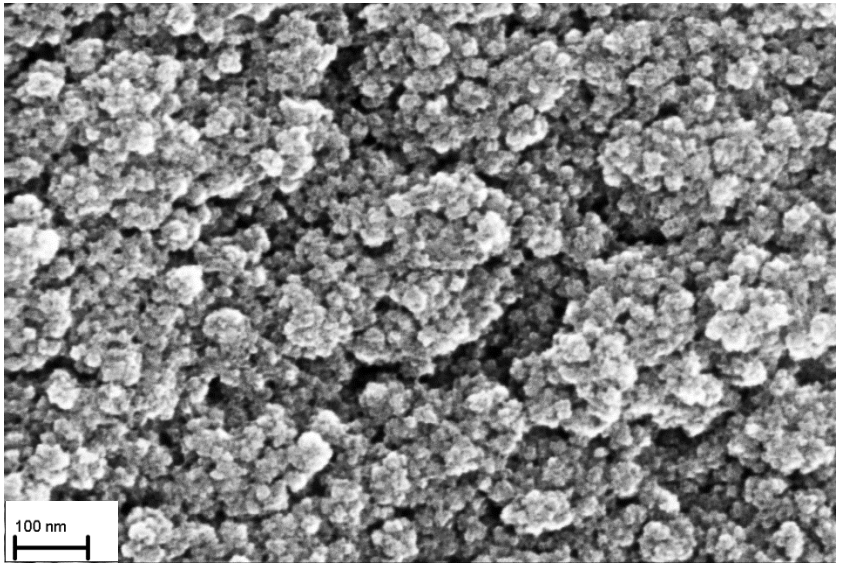
SEM is an easy task! Focus and take a “Photo”!



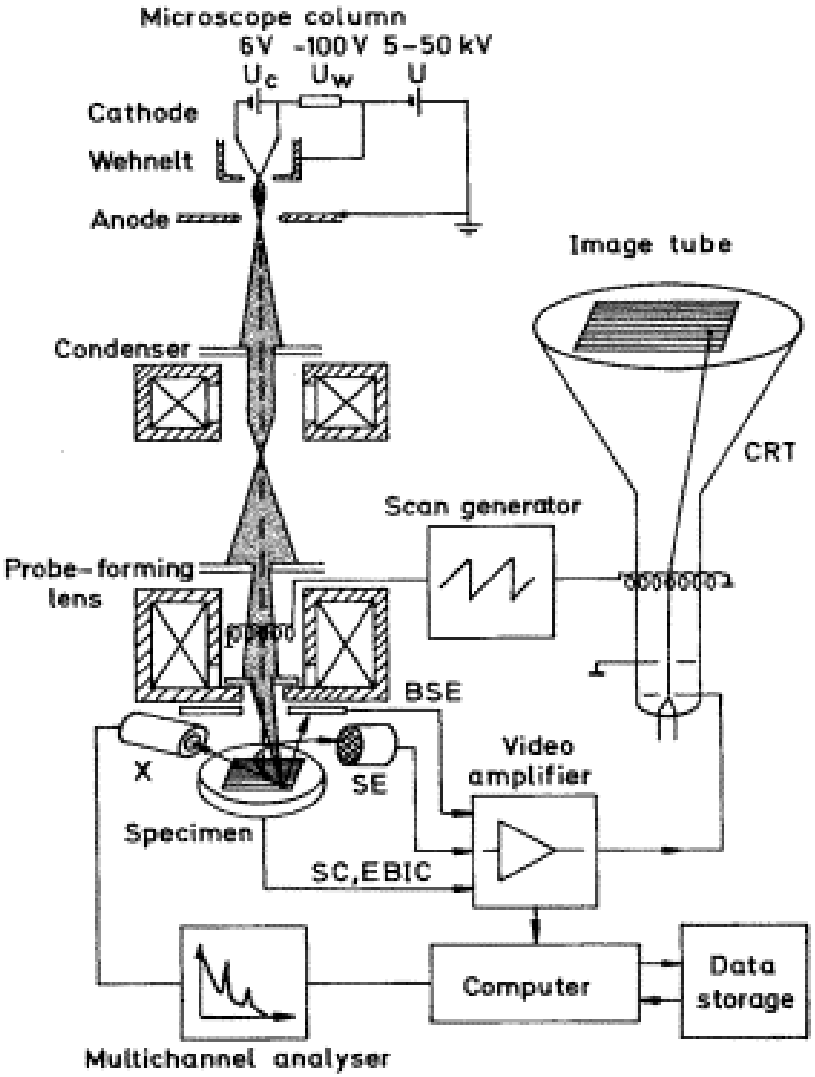
Please comment on the picture?

Any suggestion what it is?

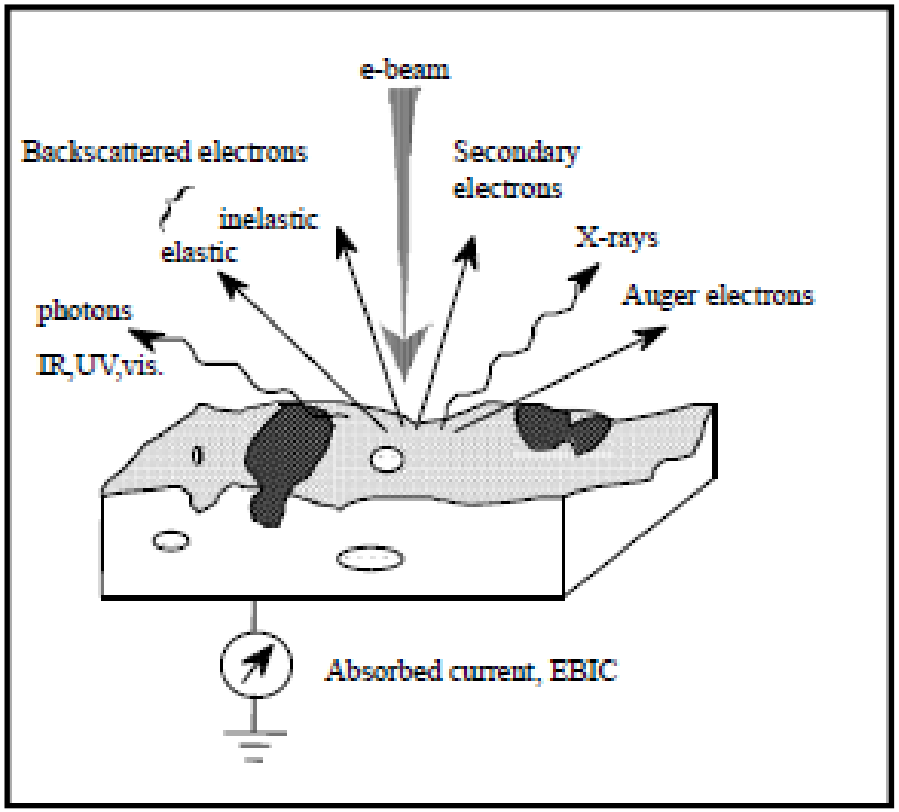
Maybe this one is more obvious?



1. SEM image formation



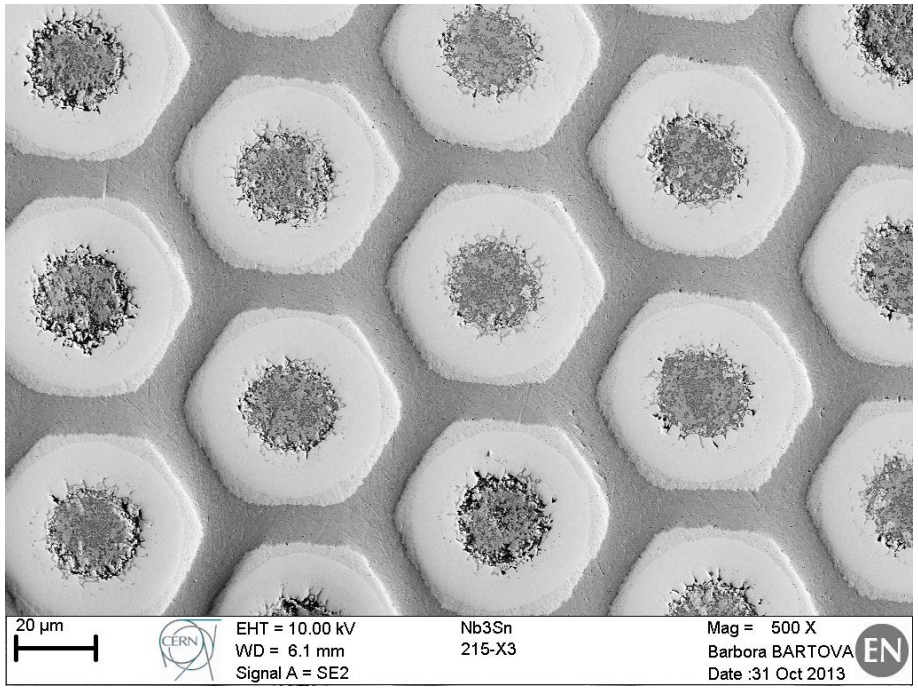
1. SEM – signals



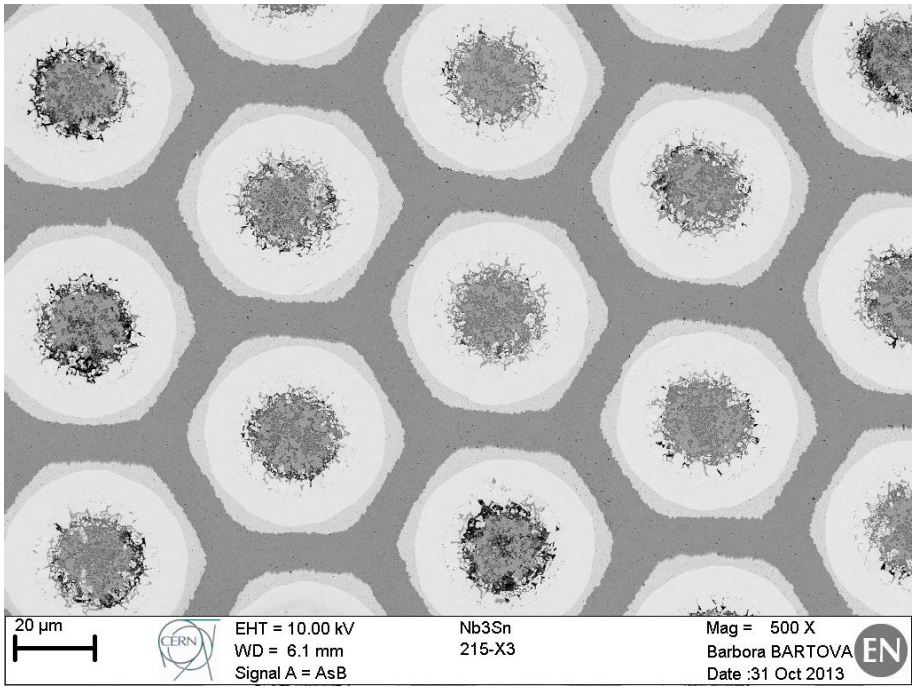
- **SE** – secondary electrons **(0-30 eV)**
- **BSE** – backscattered electrons **(>50 eV)**
- Auger electrons
- Photons – Vis, UV, IR, **X-Rays**
- Phonons, heating
- Absorption of incident electrons (EBIC current)

1. SEM – SE and BSE

SE – secondary electrons



BSE – backscattered electrons



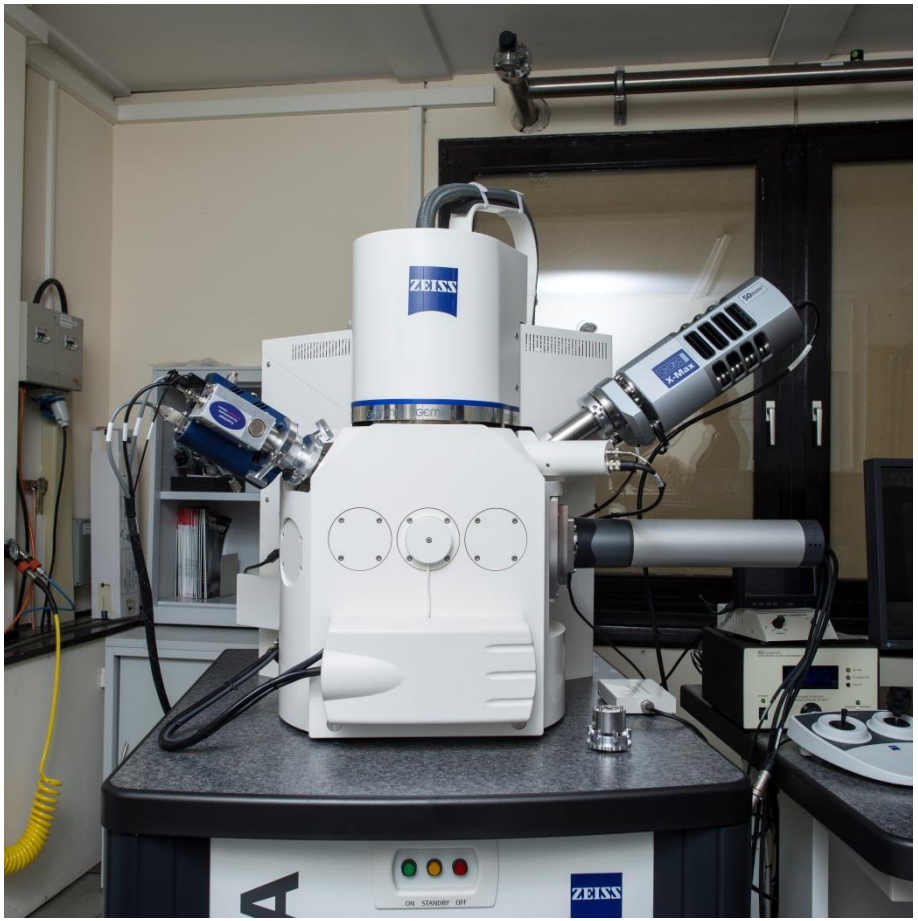
#B215 is a Powder-in-Tube (PIT) Nb3Sn wire (the Sn source is a mixture of NbSn2 and Sn powder particles inside Nb tubes). It contains 288 filaments.

- Topography
- Electrons with low energy leaving the sample surface
- Intensity depends on surface inclination

- Compositional contrast
- Electrons with high energy backscattered from bellow the surface
- Intensity depends on atomic weight

2. SEM – Typical SEM

At CERN: ZEISS SIGMA



Resolution: 1.7 nm@ 15 kV; 3 nm@1kV

Acceleration Voltage: 0.1 - 30 kV

Probe current: 4 pA – 20 nA, with 40 nA High current option

Electron source: Schottky field emitter

<u>Standard detectors:</u>
Everhart-Thornley secondary electron detector
High-efficiency In-Lens SE detector

<u>Optional detectors:</u>
BSE detector
EDS SDD INCASynergy 350 X-MAX 50
EBSD HKL Advanced Channel 5

2. SEM – Typical SEM – advantage of In-Lens detector

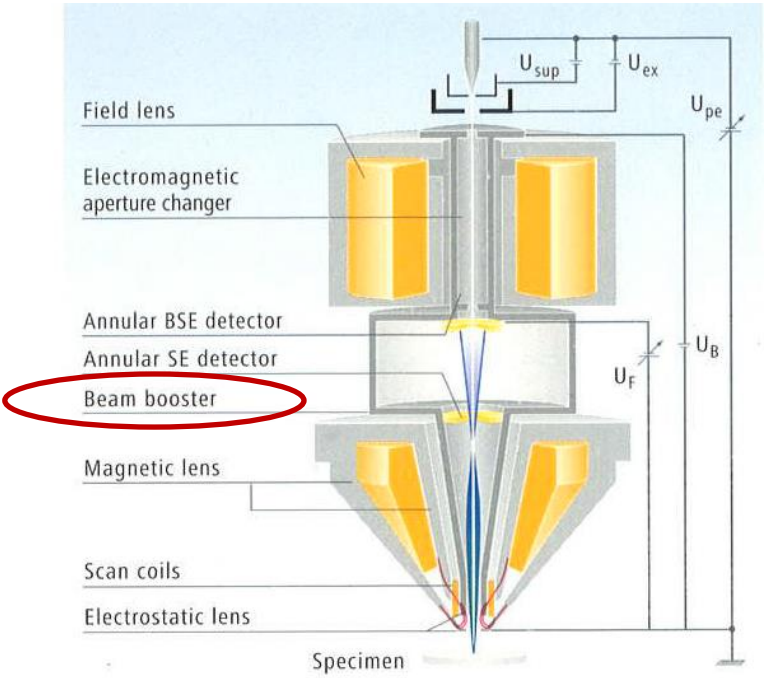
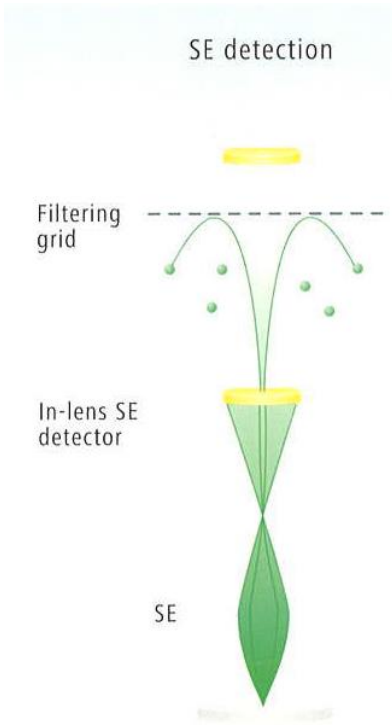


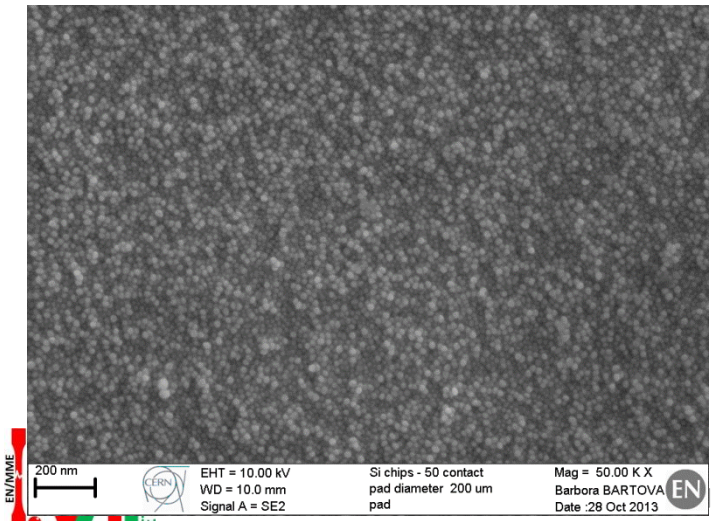
Fig. 2: GEMINI bias concept

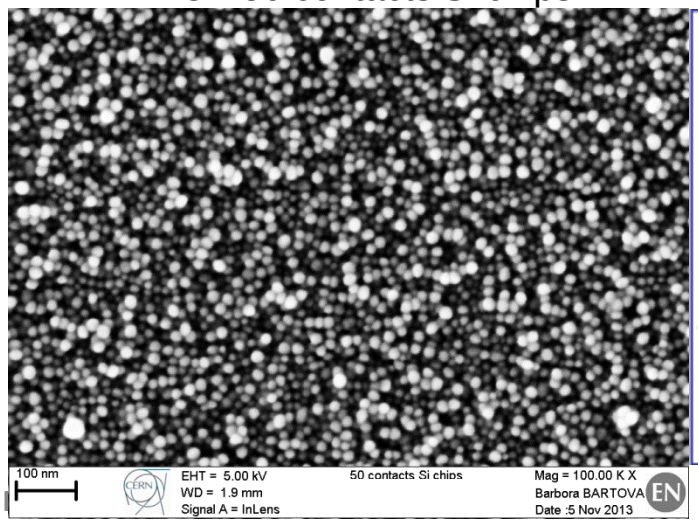


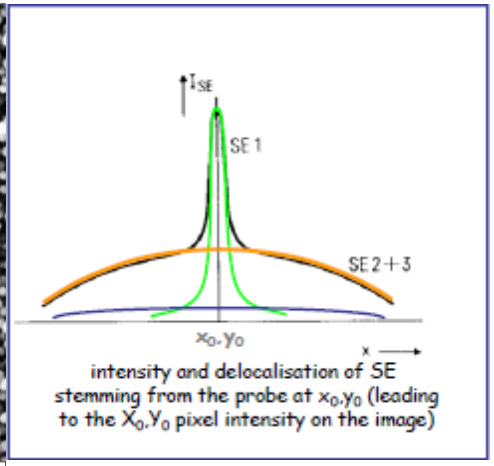
Beam booster

- attracts and accelerates SE from sample surface towards In-lens detectors
- helps to get highest resolution and beam stability

ALICE 50 contacts Si chips

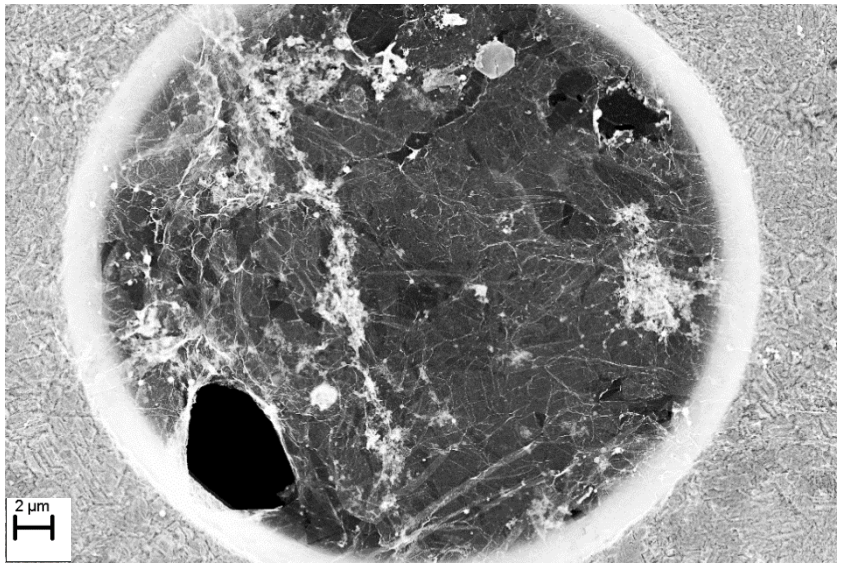


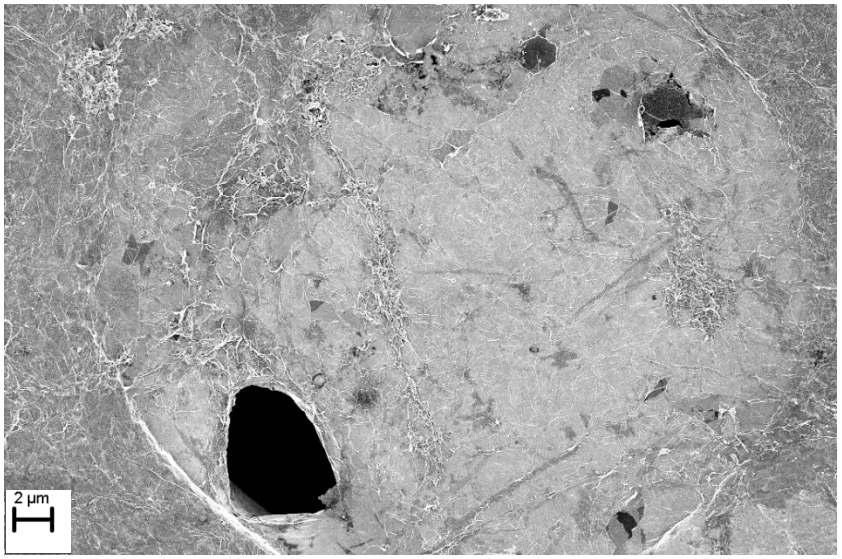




2. SEM – Typical SEM – low kV imaging

5 kV Graphene on Cu grid R&D 1 kV





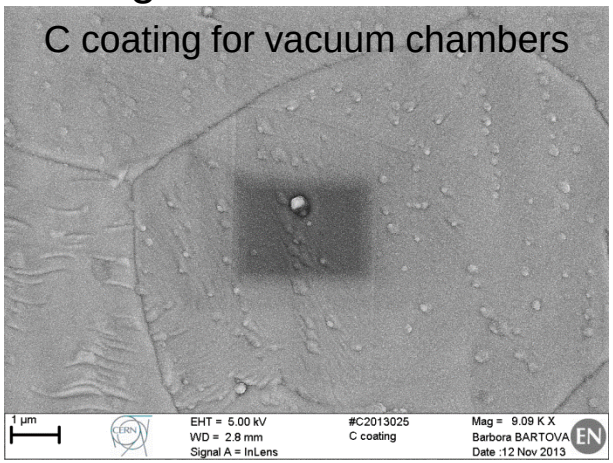
Advantages:

- Reveals real surface
- Observation of non – conductive, low density samples
- Contrast enhancement

Disadvantages:

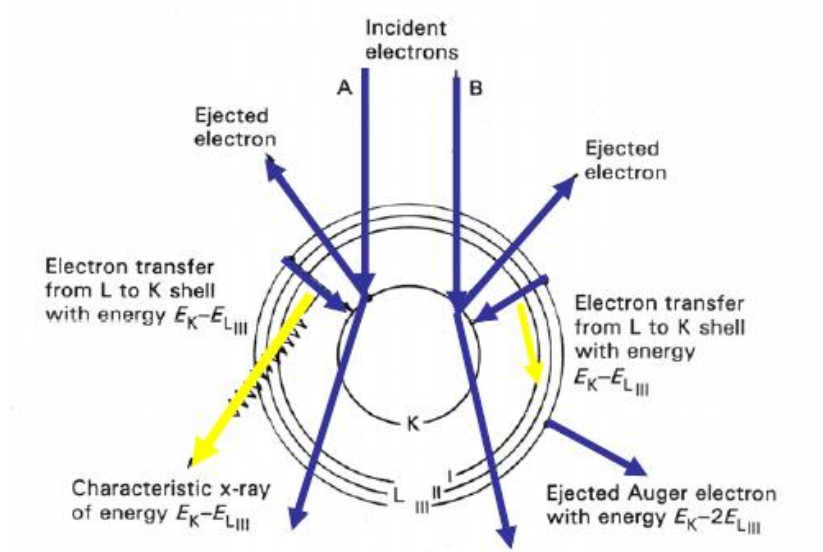
- Contamination by hydrocarbons
- Spoils low kV imaging
- ➢ Plasma cleaning of chamber and samples

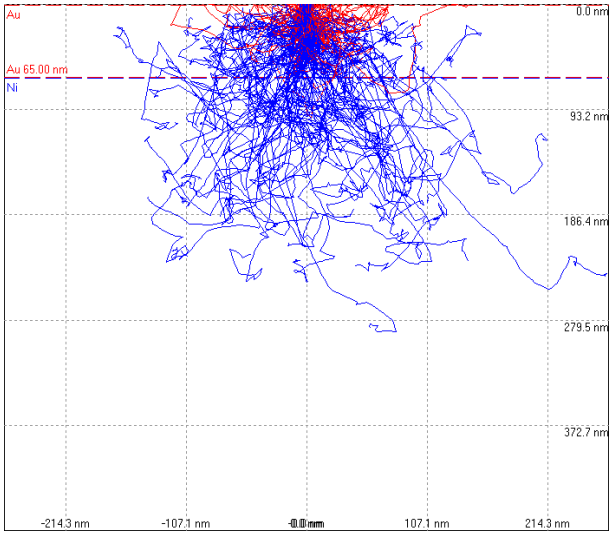
C coating for vacuum chambers

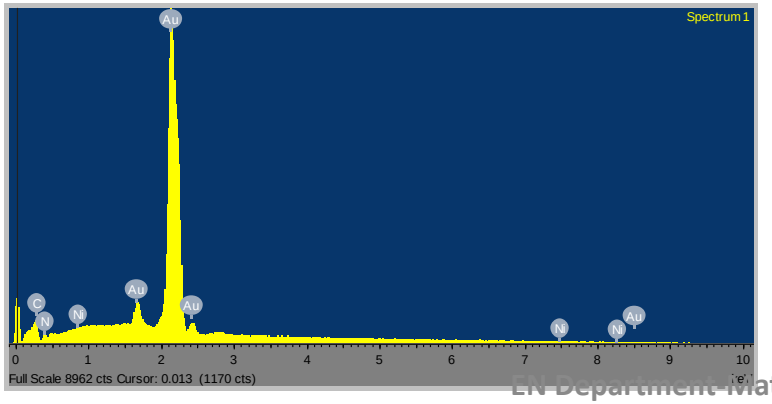


3. SEM – EDS and EBSD

- Generation of X-rays
- Detection by SDD detector
- Quantification by commercial software, Interaction volume Monte Carlo simulation







Blue: scattered electrons
Red: back scattered electrons – leave sample surface

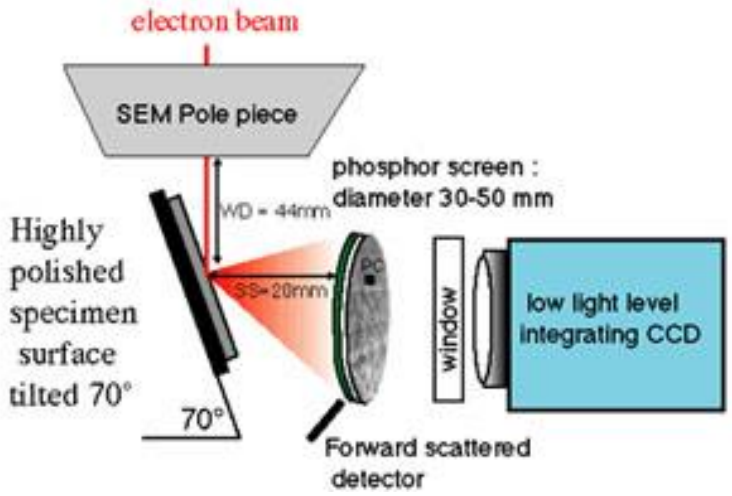
3. SEM – EDS and EBSD

Electron backscatter diffraction is an SEM based microstructural-crystallographic technique to measure the crystallographic orientation.

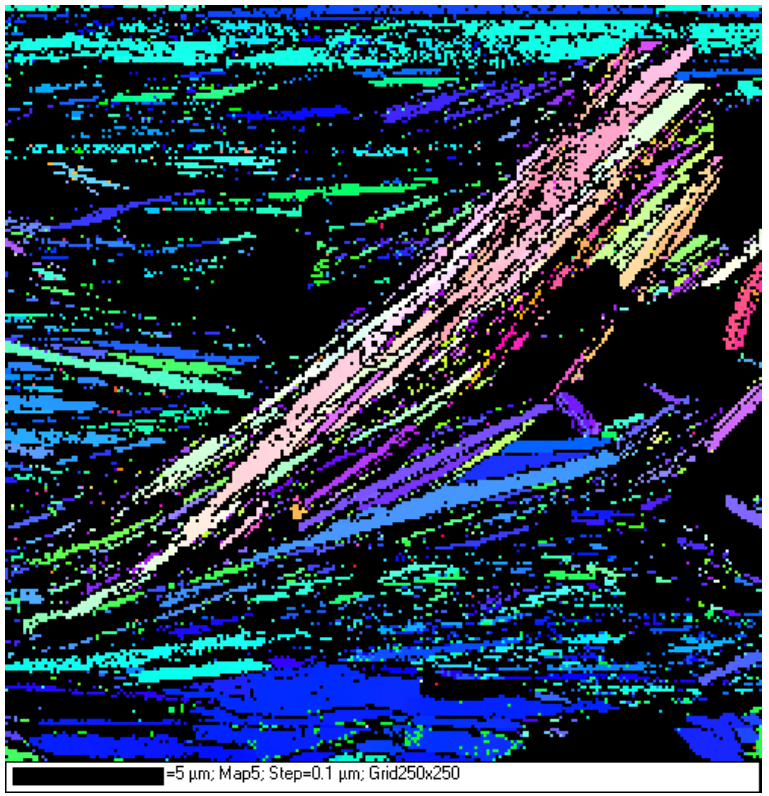
electron beam : 15-25 kV, 0.01-50 nA

working distance (WD) : 8 - 45 mm
determines spatial resolution
and max. specimen area

specimen to screen distance (SS): 10-40 mm
determines solid angle (60 - 90°)
large angles->short SS

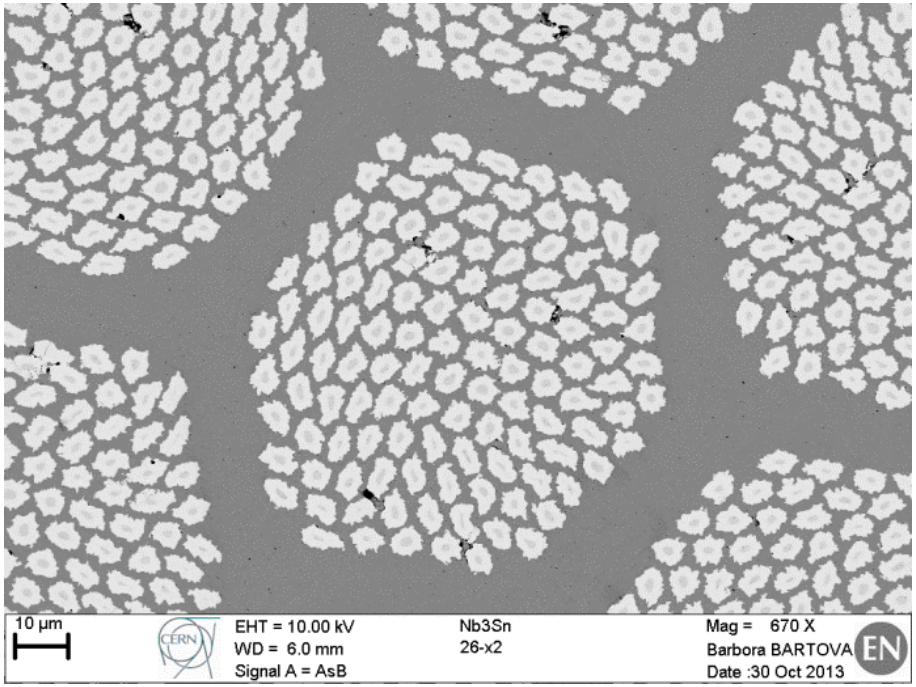


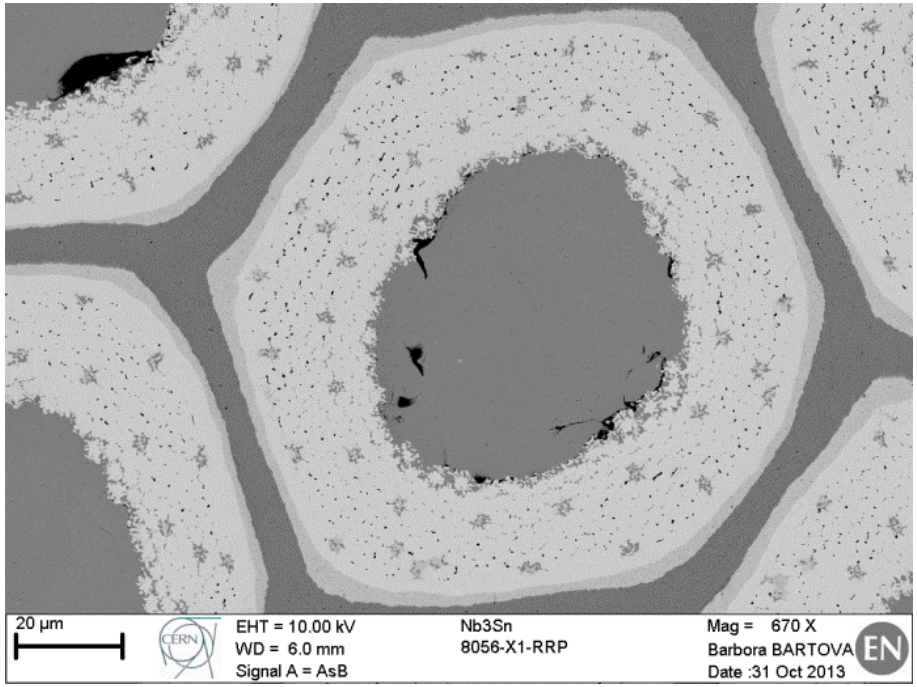
High temperature BSCCO superconducting magnet



4. SEM – Materials analysed at CERN

Application is for high field magnets operating at 4.2 K or 1.9 K





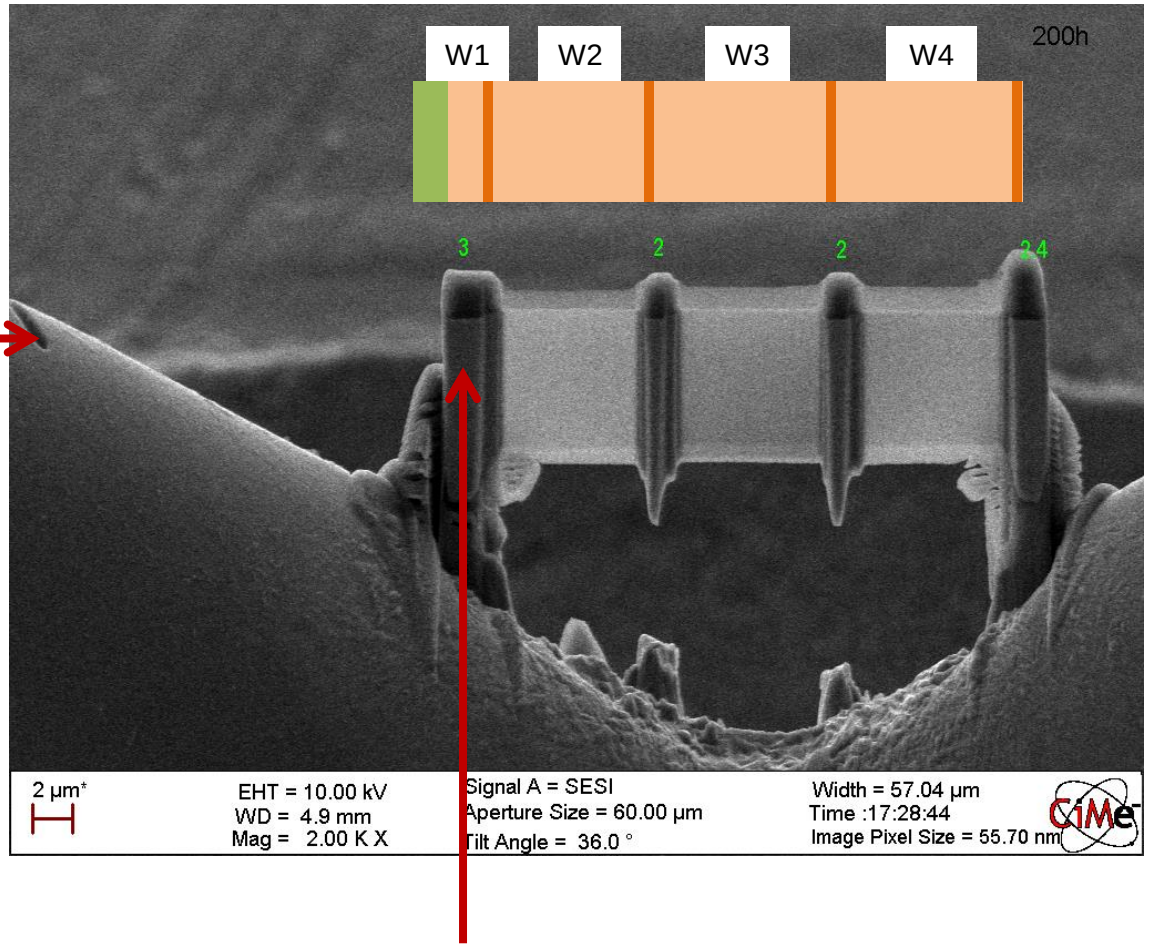
#26 is a bronze route Nb3Sn wire (Nb filaments are in a bronze matrix, and during a heat treatment Sn from the matrix diffuses into the Nb filaments forming Nb3Sn). It has more than 14000 filaments.

#8056 is Restacked Rod Process (RRP) Nb3Sn strand (pure Sn is inside the Cu matrix). It has 60 filaments.

4. SEM – Materials analysed at CERN

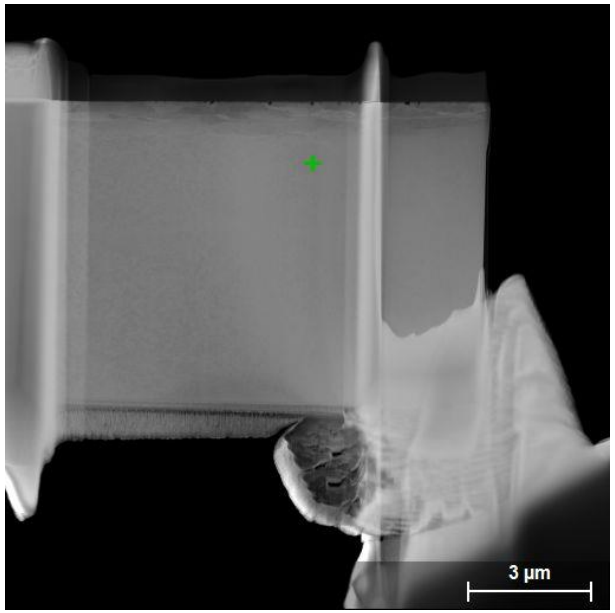
A Nb3Sn strand studied for ITER applications suffered from decrease in RRR of Cu (residual resistivity ratio) – Cr from plating diffuses to Cu matrix – TEM measurements @ EPFL

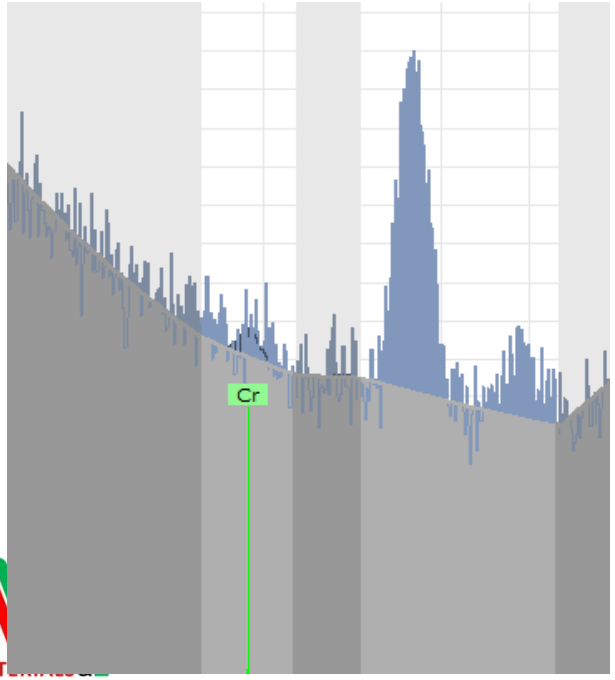
Cr plating side



After first observation by TEM the first part of lamella was thinned as well.

4. SEM – Materials analysed at CERN





Quantification results
Atomic percent (%)

Date: 06.02.2013

Spectrum	S	Cr	Cu
1 W1-s1	0	0	100
2 W2-s1	0	0.015784	99.98422
3 W2-s2	0	0.029017	99.97098
4 W2-s3	0	0.015008	99.98499
5 W3-s1	0	0.019895	99.9801
6 W3-s2	0	0.009489	99.99051
7 W3-s3	0	0.005492	99.99451
8 W3-s4	0	0	100
9 W3-s5	0	0	100
10 W4-s1	0	0	100
11 W4-s2	0	0	100
12 W4-s3	0	0	100
13 W4-s4	0	0	100
Mean value:	0	0.007284	99.99272
Sigma:	0	0.009759	0.009759
Sigma mean:	0	0.002707	0.002707

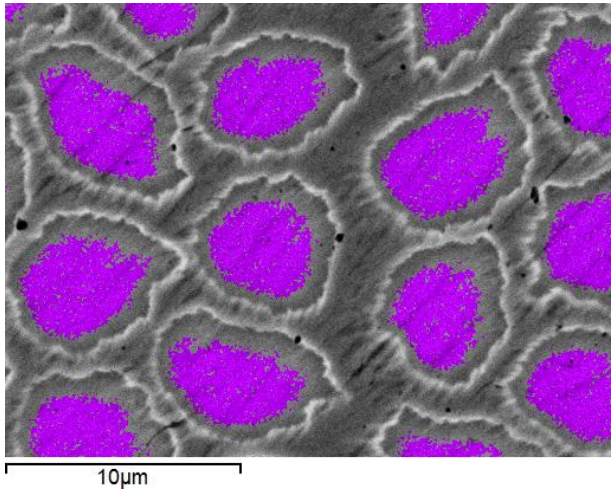
4. SEM – Materials analysed at CERN

NbTi superconducting magnet used in LHC

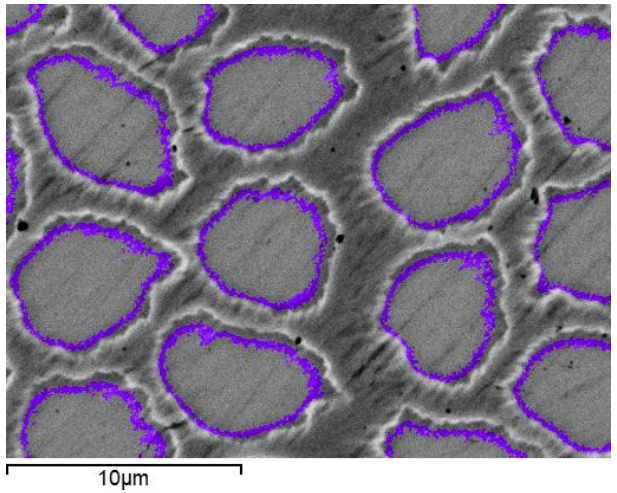
Quant maps show that there is an area of diffusion of Cu in Nb-Ti phase and other way around.

Spectrum	O	Ti	Cu	Nb	Total
wt.%					
Phase01	3.1	42.4	1.8	52.7	100.0
Phase02	2.2	33.7	17.7	46.4	100.0
Phase03	1.9	23.6	35.1	39.4	100.0
Phase04	1.8	7.5	72.1	18.7	100.0
Phase05	1.0	2.4	91.9	4.8	100.0

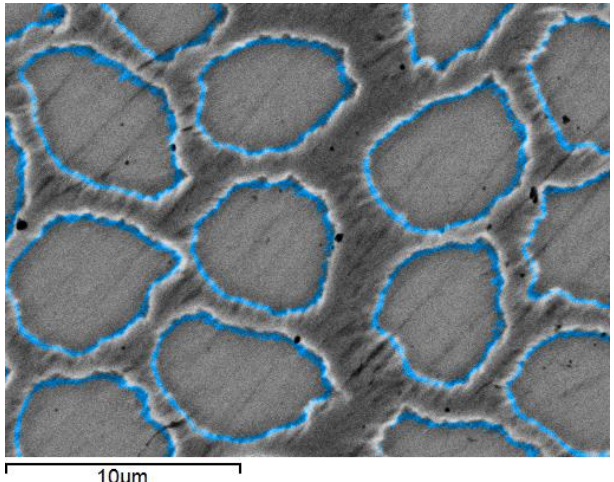
Phase 01



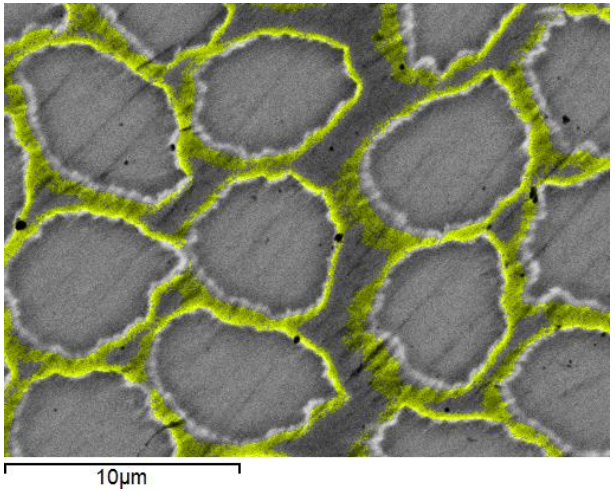
Phase 02



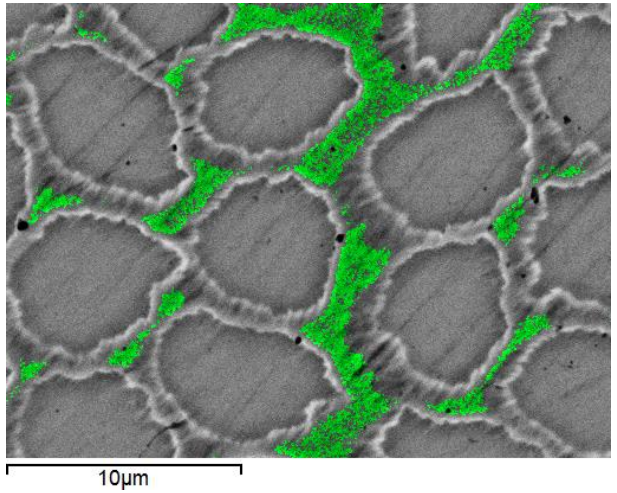
Phase 03



Phase 04



Phase 05



4. SEM – Materials analysed at CERN

Development of NEG coating for vacuum chambers – control of grain size, morphology and chemical composition.

TiZrV NEG coating

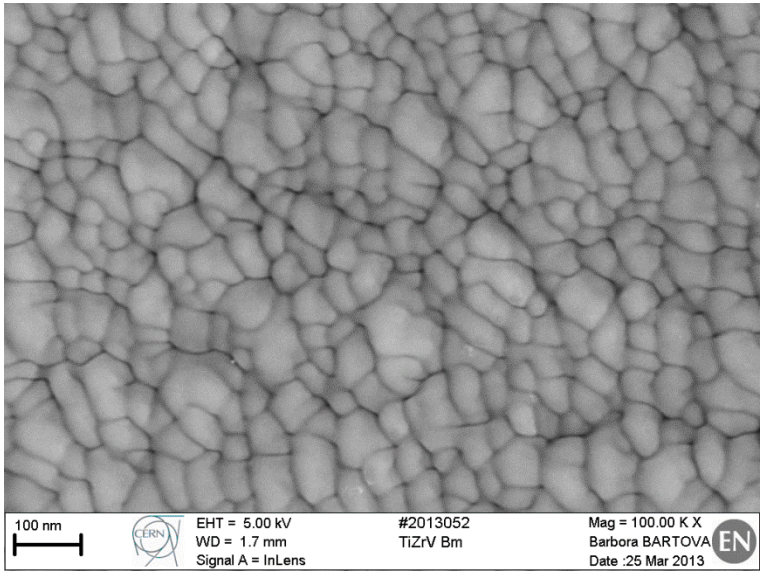
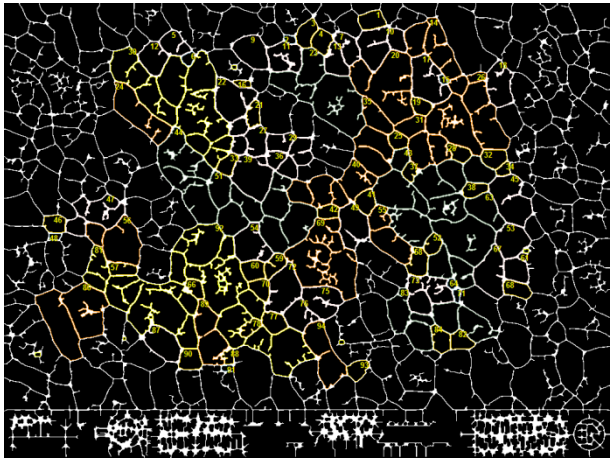
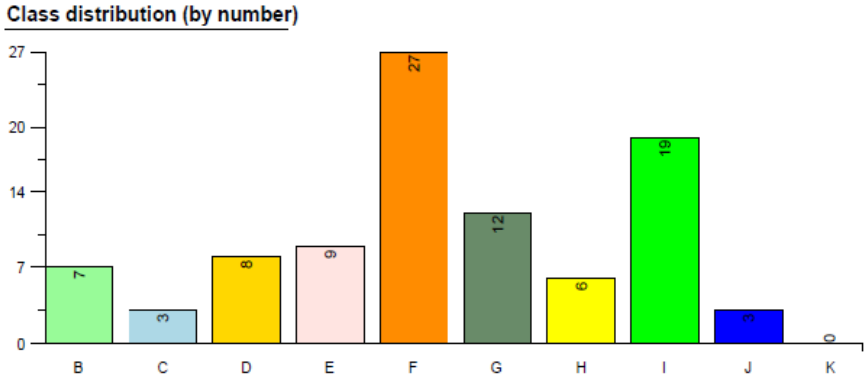


Image analysis with software of optical microscope





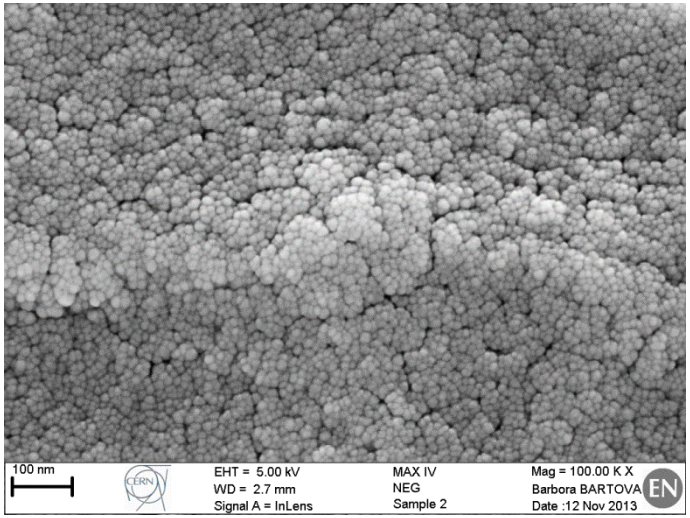
Particle size distribution

Class	>	<=	Absolute number of particles	Particles allowed	Status
B	0	10	7	1920	OK
C	10	20	3	1920	OK
D	20	30	8	960	OK
E	30	40	9	960	OK
F	40	60	27	480	OK
G	60	80	12	240	OK
H	80	100	6	120	OK
I	100	200	19	120	OK
J	200	500	3	120	OK
K	500		0	120	OK

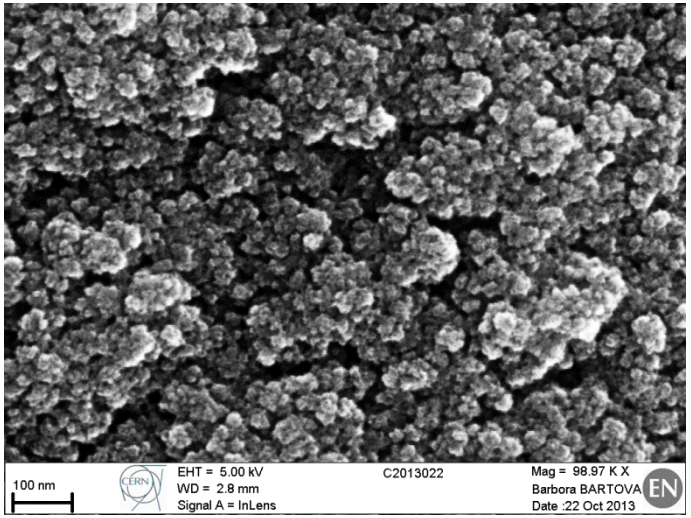
4. SEM – Materials analysed at CERN

Development of NEG coating for vacuum chambers – control of grain size, morphology and chemical composition.

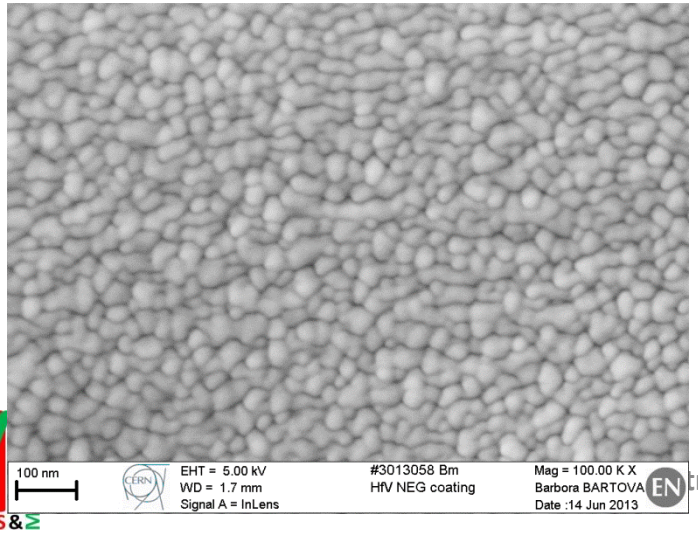
TiZrV NEG coating



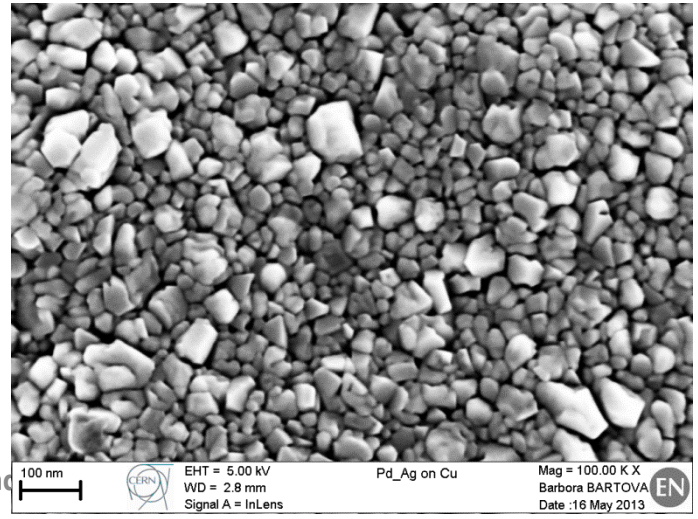
C coating



HfV NEG coating

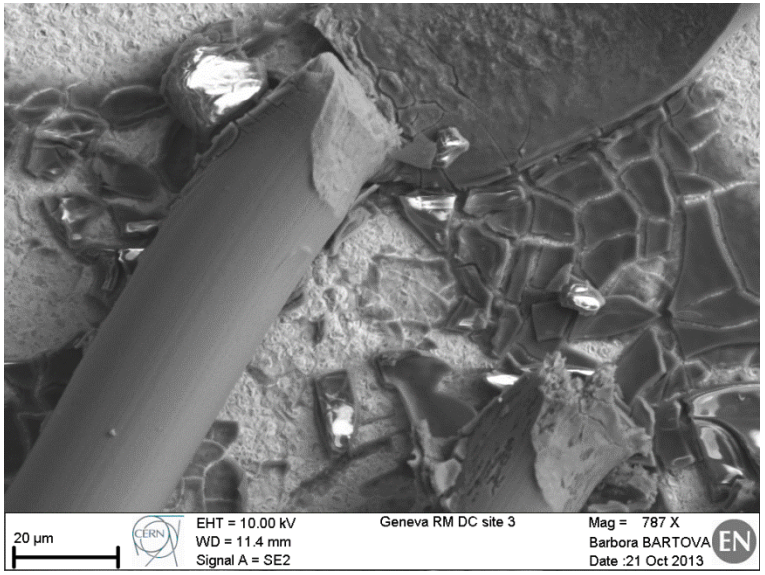


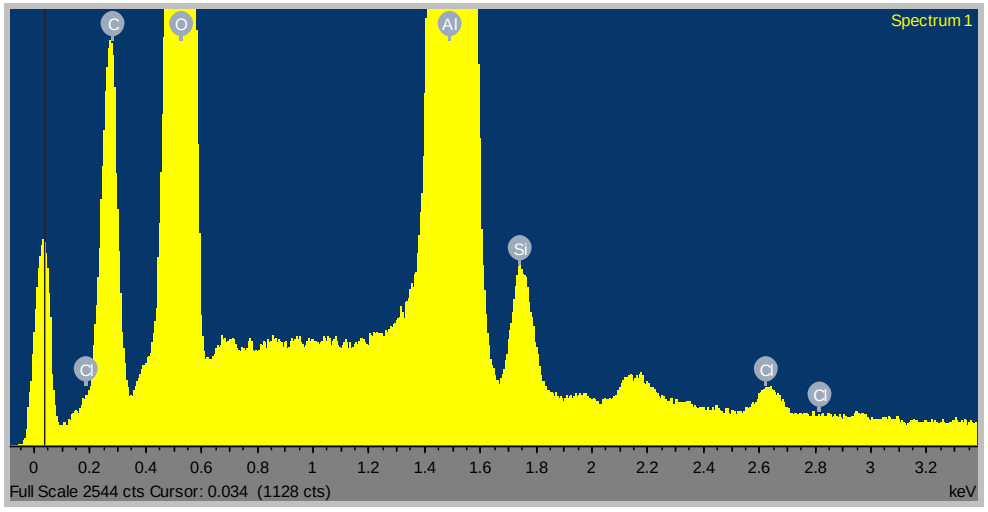
Pd coating



4. SEM – Materials analysed at CERN

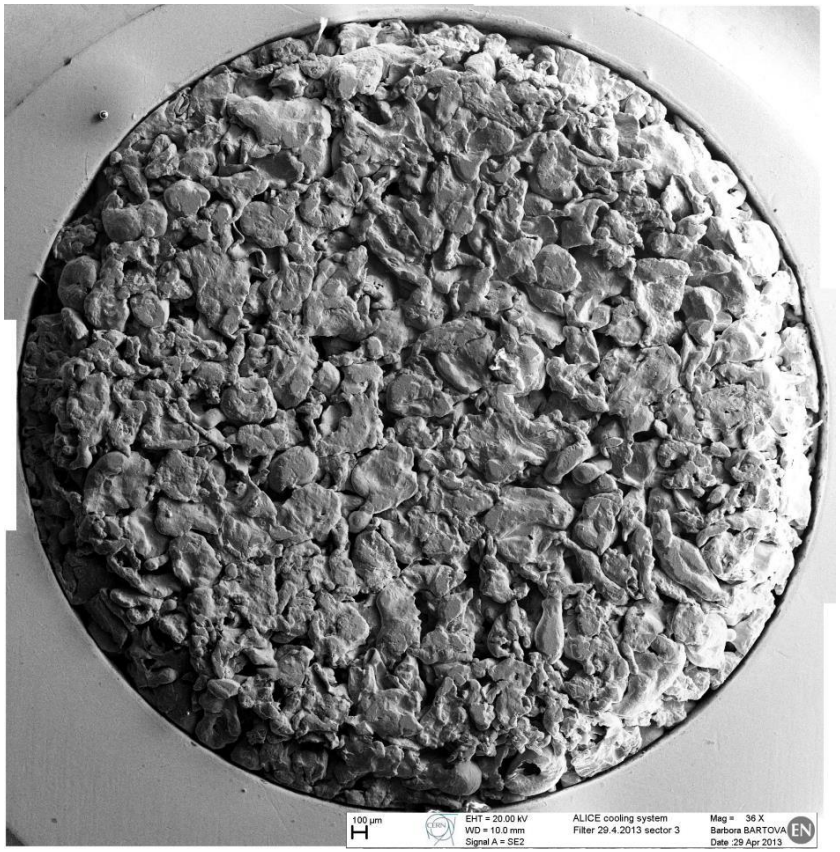
ATLAS Insertable B-Layer – detection of local corrosion

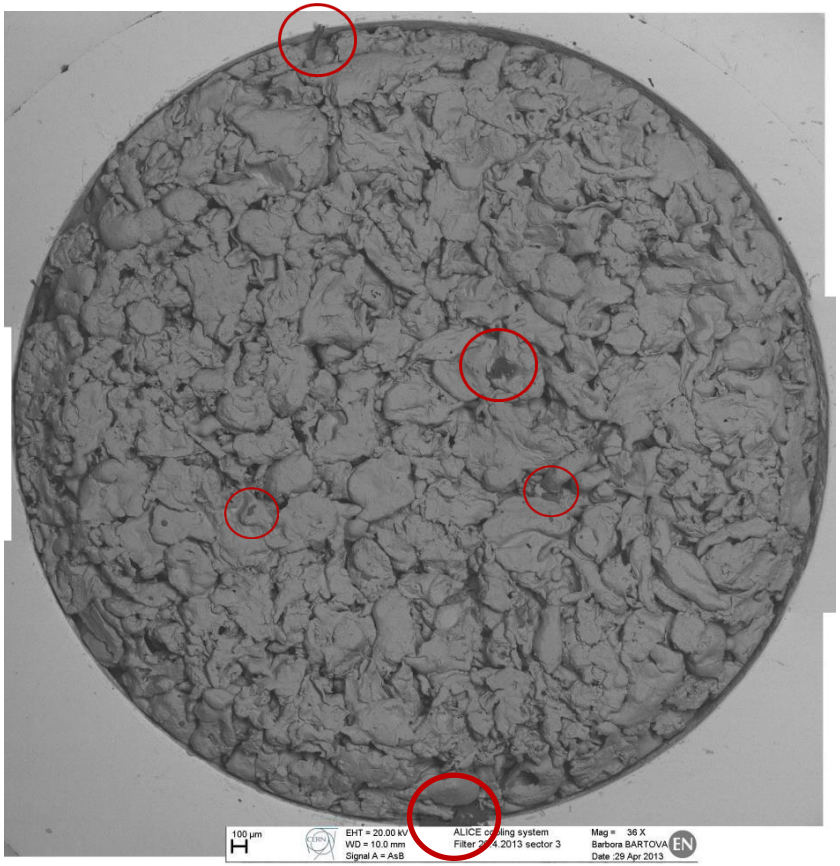




4. SEM – Materials analysed at CERN

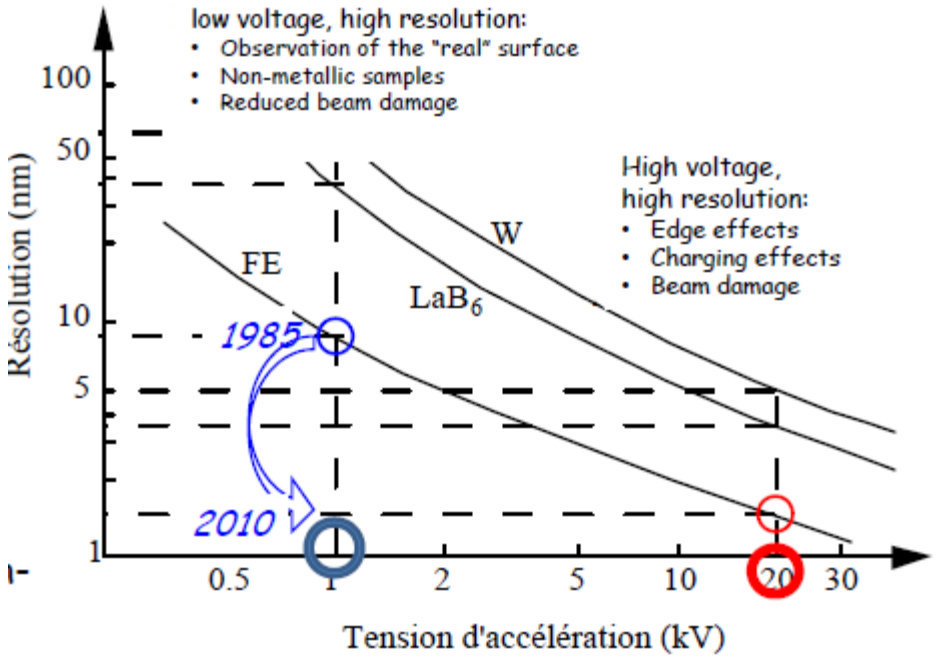
ALICE cooling system – drilling and control of filters





CONCLUSIONS

Detail materials analysis thanks to new generation of SEM



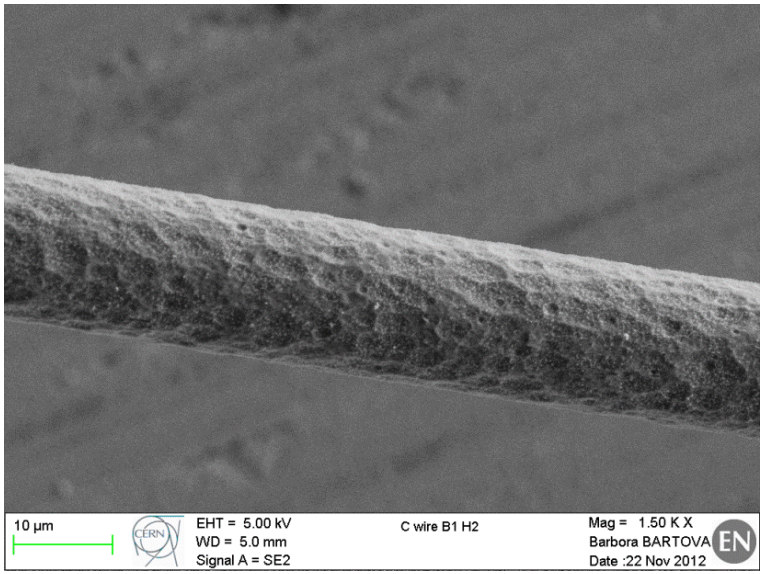
Field emission gun
Monochromators
Beam booster, deceleration
New generation of detectors
Lens design

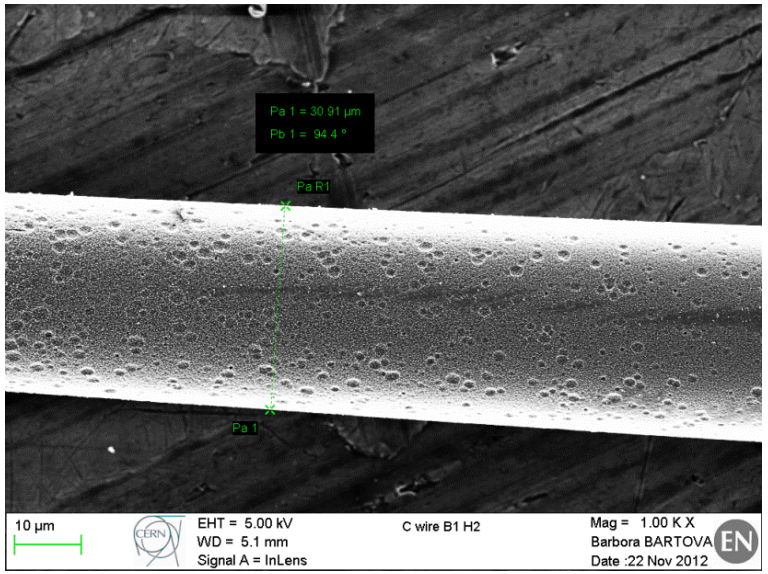
Analytical SEM
SDD EDX detectors
High speed EBSD detectors
High beam current

Thank you for your attention!

4. SEM – Materials analysed at CERN

Degradation of C wire for the beam measurements





Particles – cryogenics installation

Si chip with in print