Comparison of the same Mo coating on different substrates by SEM. Effect on the resistivity

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HiColDEM meeting 30-08-2018

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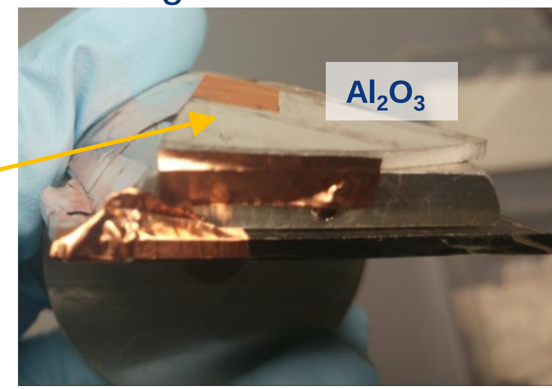
Observed specimens (SEM)

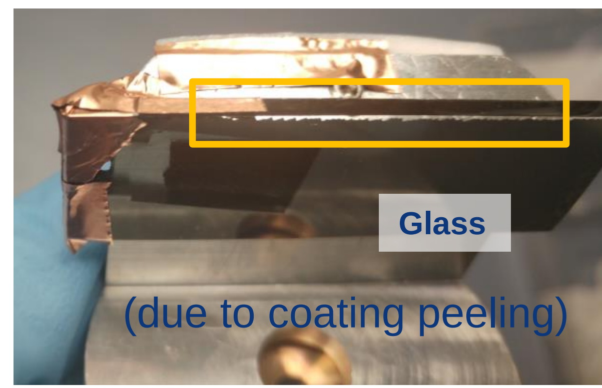
Four samples observed, Mo on:

- Glass
- Alumina
- MoGr NA-8304Gb
- CFC FS140 2800°C

Coating fracture surfaces

Observations 8-Aug-2018



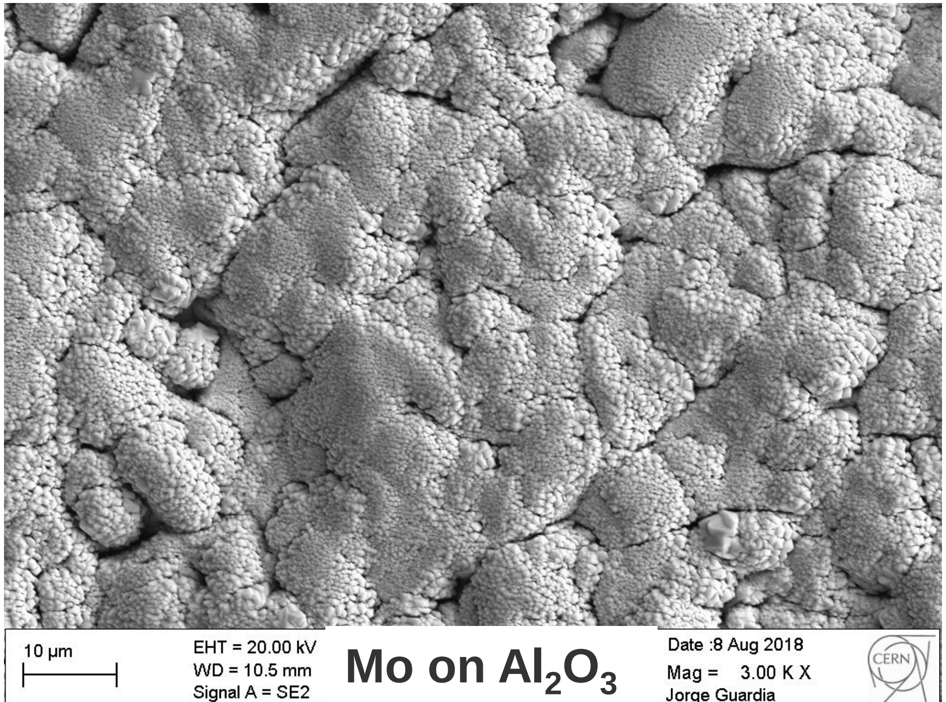


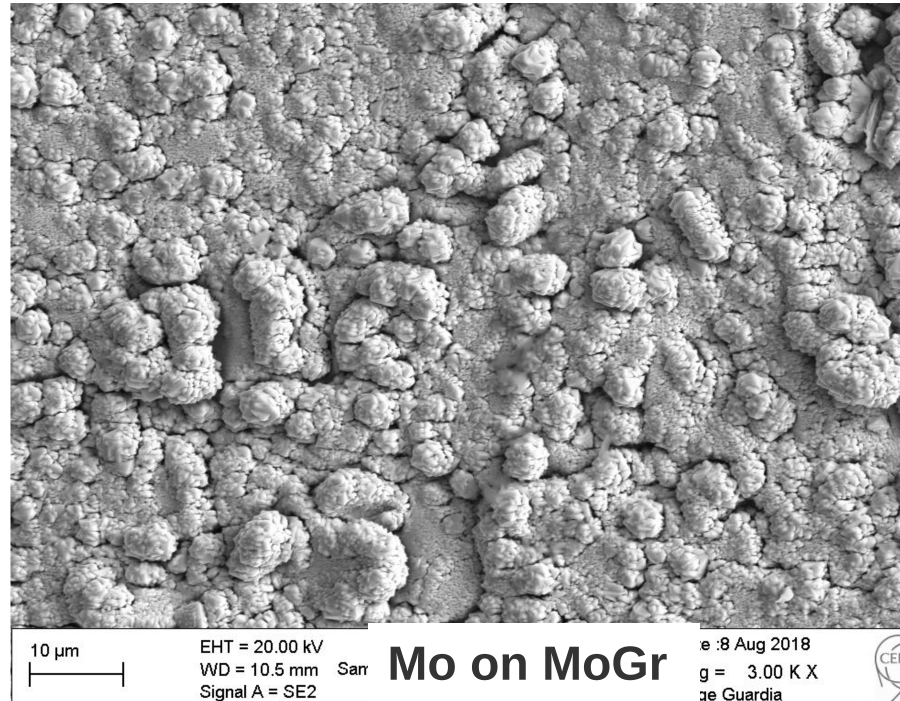
Observations 14-Aug-2018

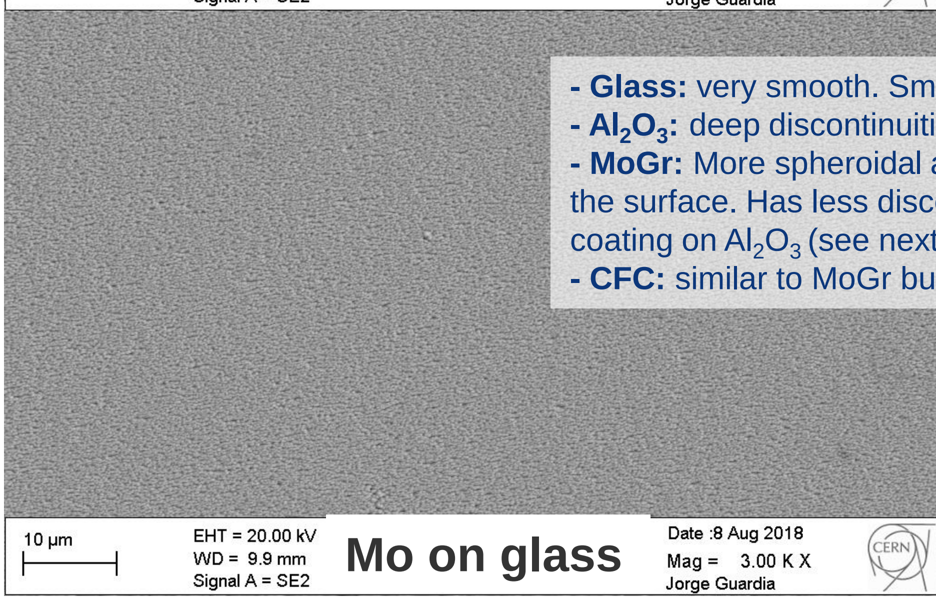
MoGr substrate observed on September 2017: Grade MG-6403Fc

Comparison all substrates

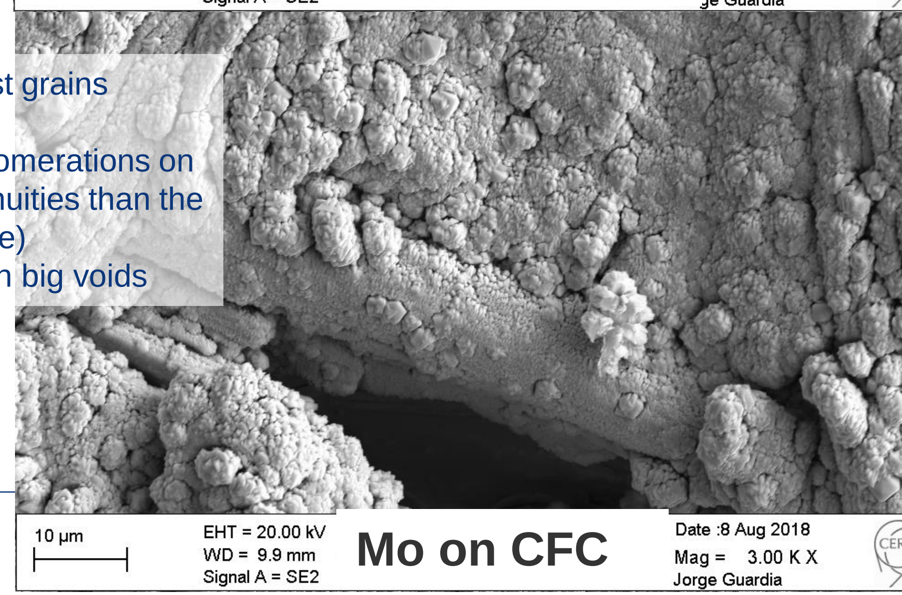
Top-view comparison. Same scale



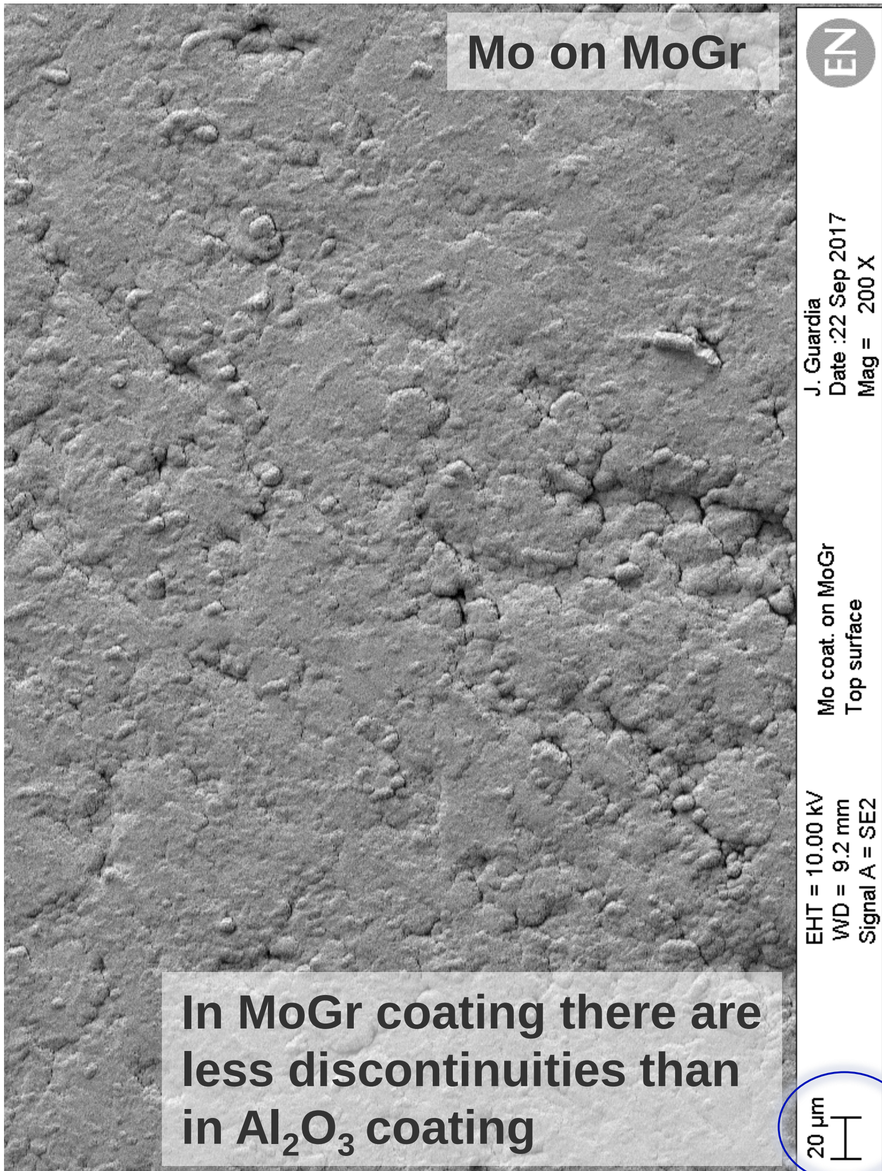


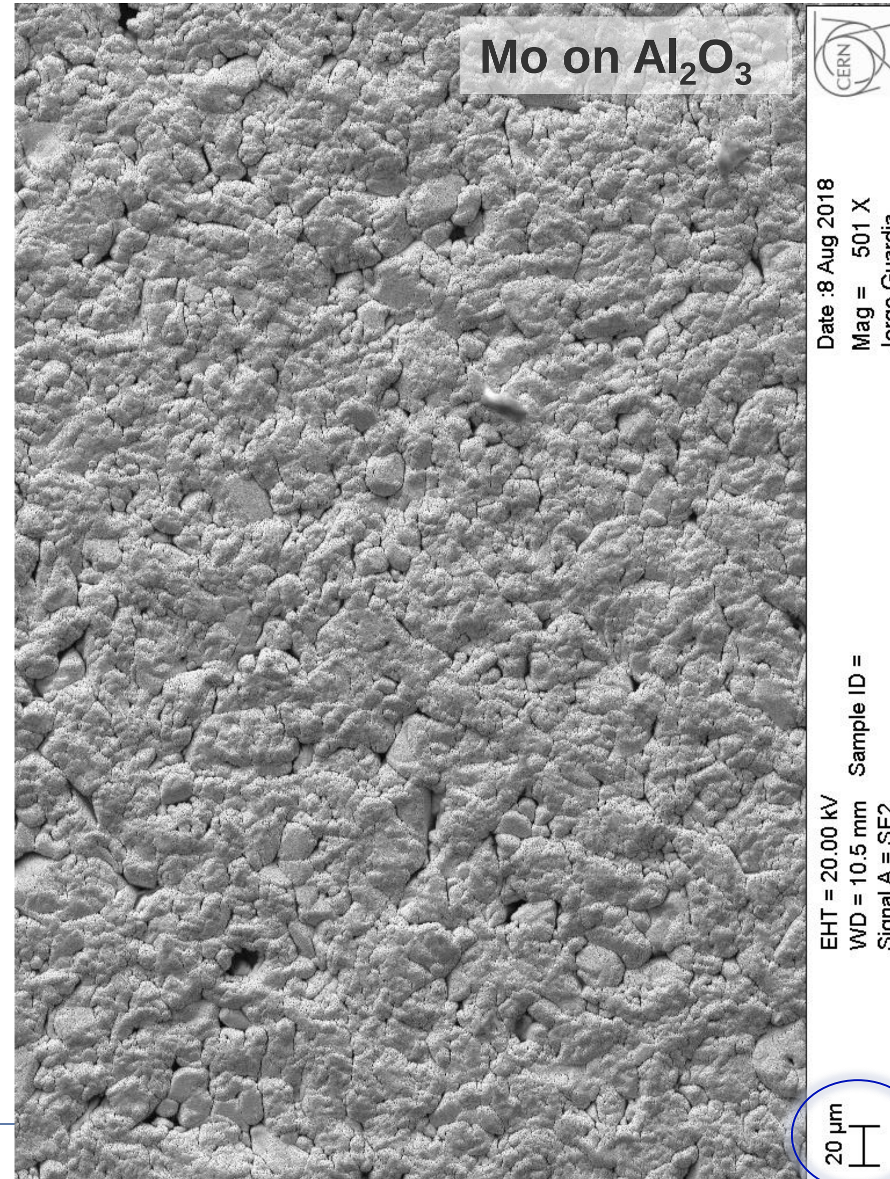


- **Glass:** very smooth. Smallest grains
- **Al_2O_3:** deep discontinuities
- **MoGr:** More spheroidal agglomerations on the surface. Has less discontinuities than the coating on Al_2O_3 (see next slide)
- **CFC:** similar to MoGr but with big voids



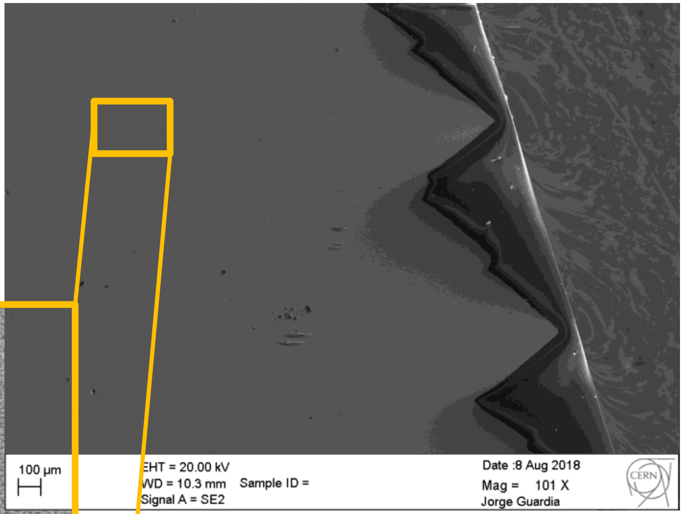
Comparison MoGr-Al_2O_3 substrates. Same scale



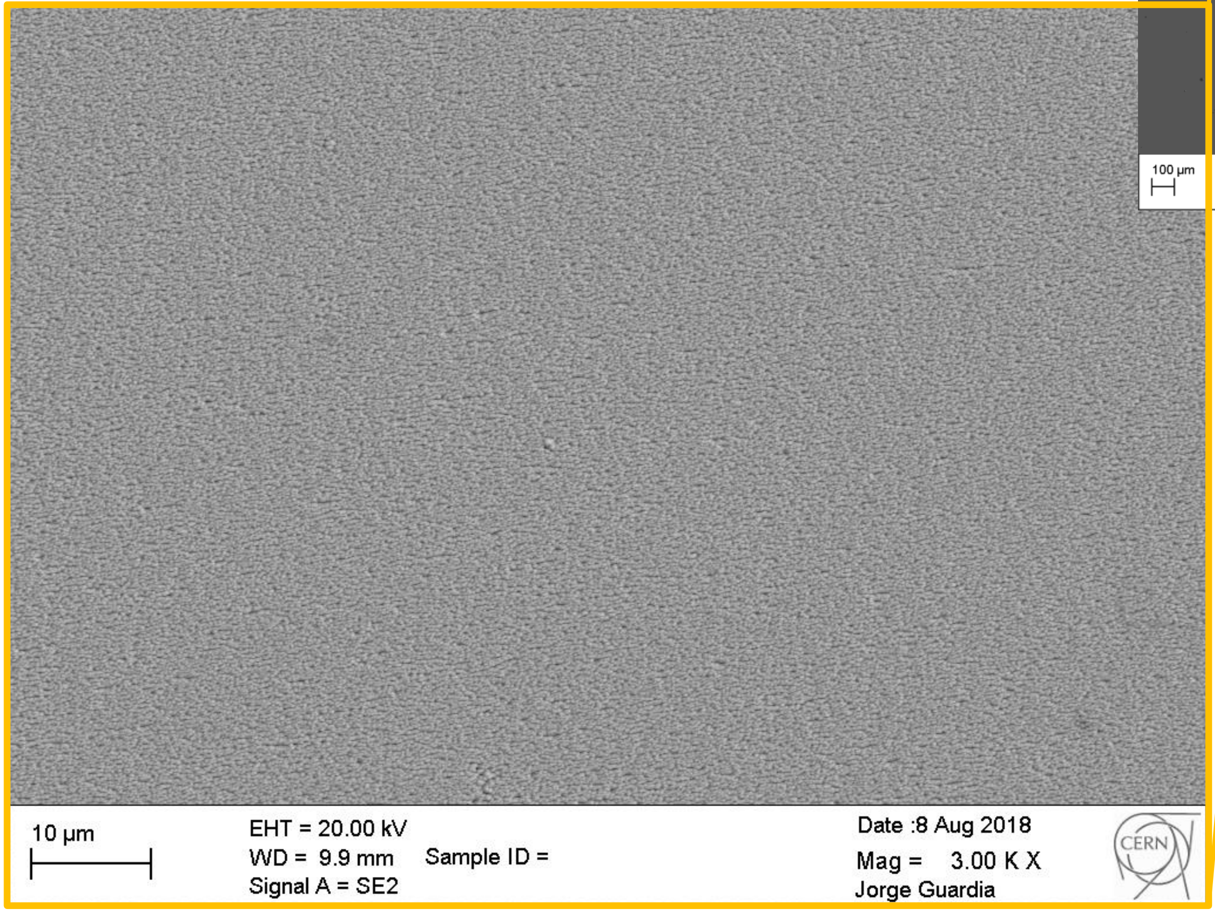


Mo on Glass

Mo on Glass

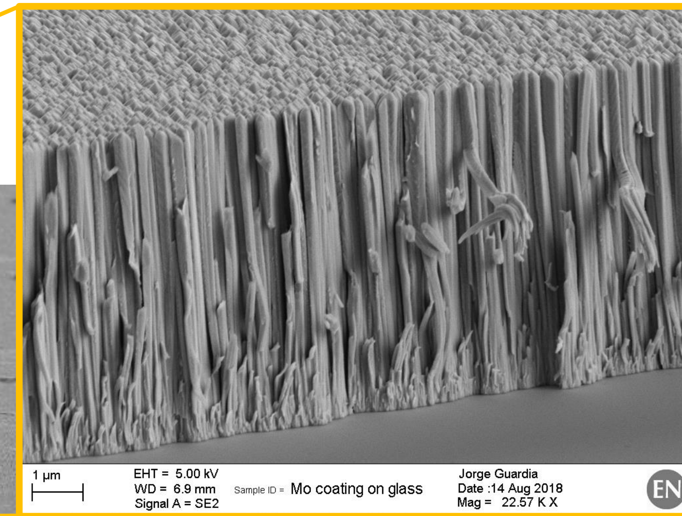


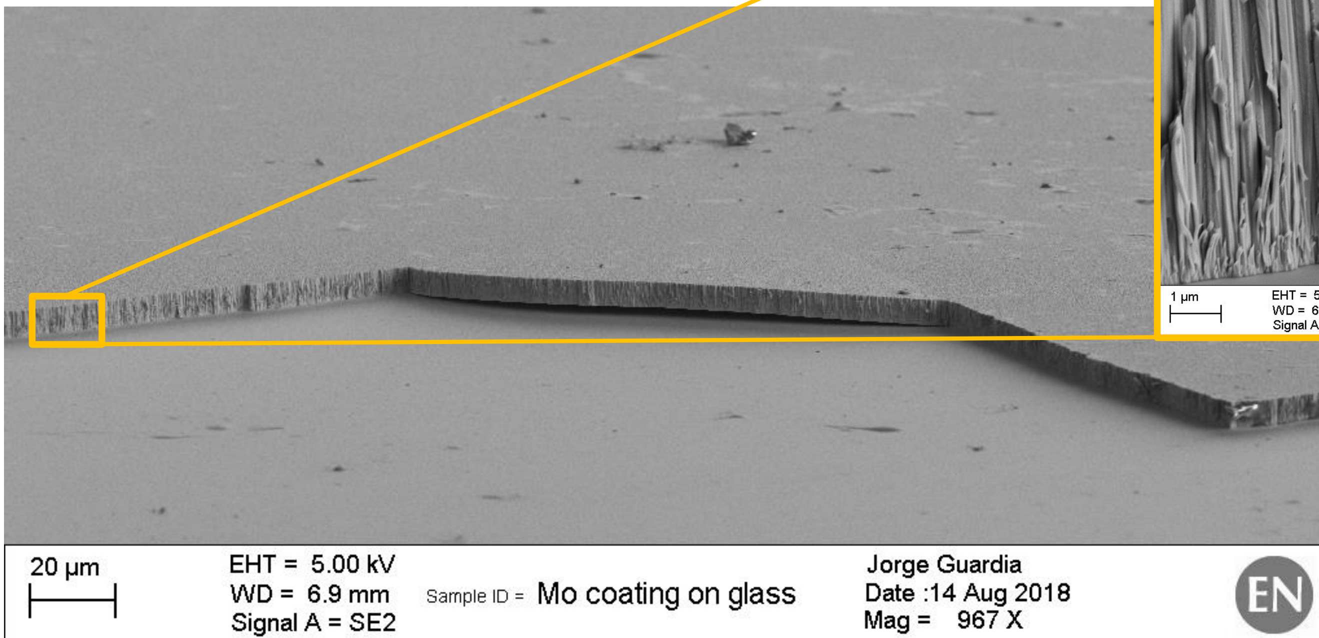
100 µm
EHT = 20.00 kV
WD = 10.3 mm
Sample ID =
Signal A = SE2
Date :8 Aug 2018
Mag = 101 X
Jorge Guardia
CERN

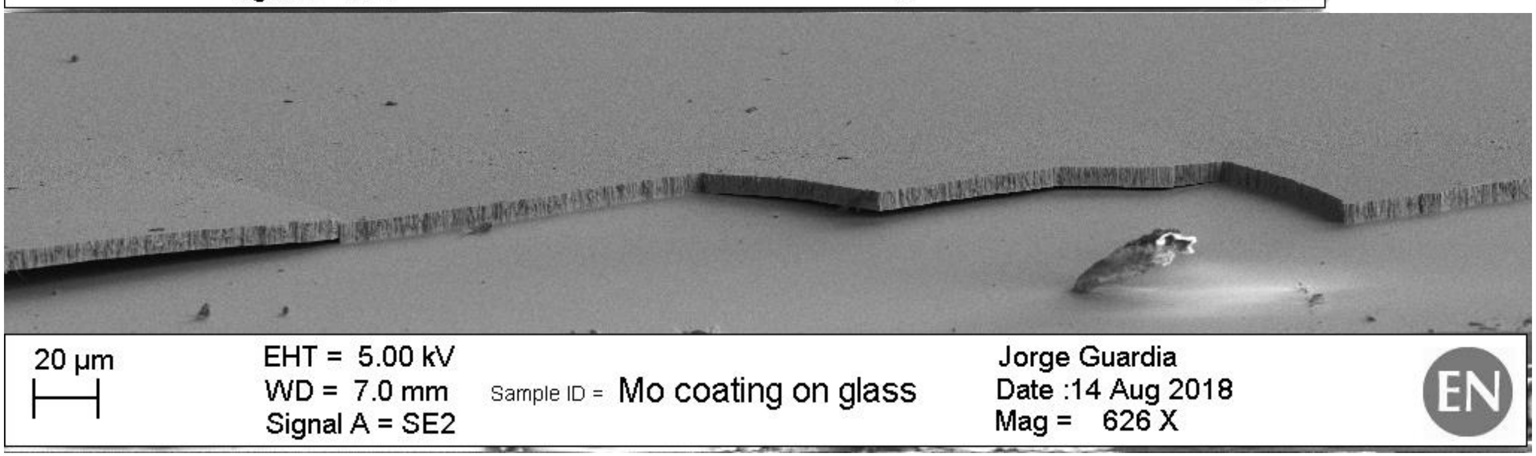


10 µm
EHT = 20.00 kV
WD = 9.9 mm
Sample ID =
Signal A = SE2
Date :8 Aug 2018
Mag = 3.00 K X
Jorge Guardia
CERN

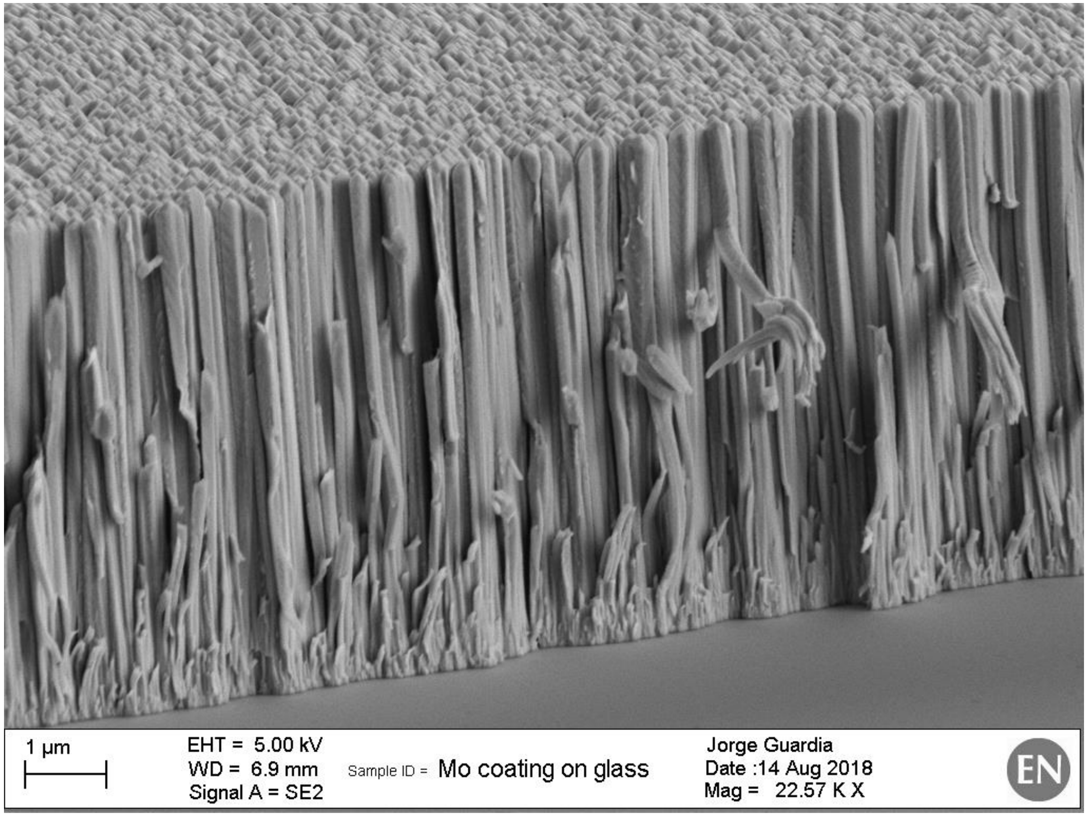
Mo on Glass

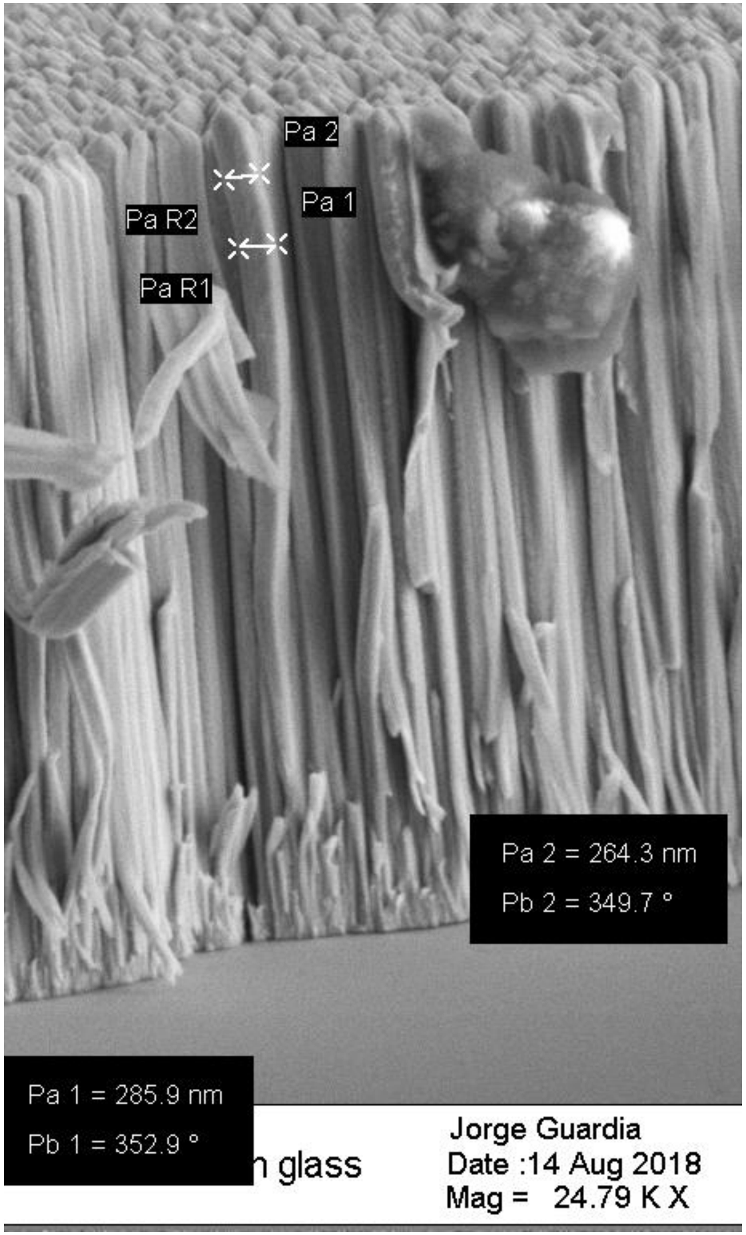

1 µm
EHT = 5.00 kV
WD = 6.9 mm
Signal A = SE2
Sample ID = Mo coating on glass
Jorge Guardia
Date :14 Aug 2018
Mag = 22.57 K X
EN


20 µm
EHT = 5.00 kV
WD = 6.9 mm
Signal A = SE2
Sample ID = Mo coating on glass
Jorge Guardia
Date :14 Aug 2018
Mag = 967 X
EN


20 µm
EHT = 5.00 kV
WD = 7.0 mm
Signal A = SE2
Sample ID = Mo coating on glass
Jorge Guardia
Date :14 Aug 2018
Mag = 626 X
EN

Mo on glass





The measured grain has a maximum lateral size of 286nm (close to the surface). This smaller than on the other substrates

The average thickness of the coating on glass was 6.05µm. This value matches the expected one (6µm).

The bottom area of the coating shows even smaller grains → changes in resistivity depending on the height. This happens in all substrates (nucleation of grains)

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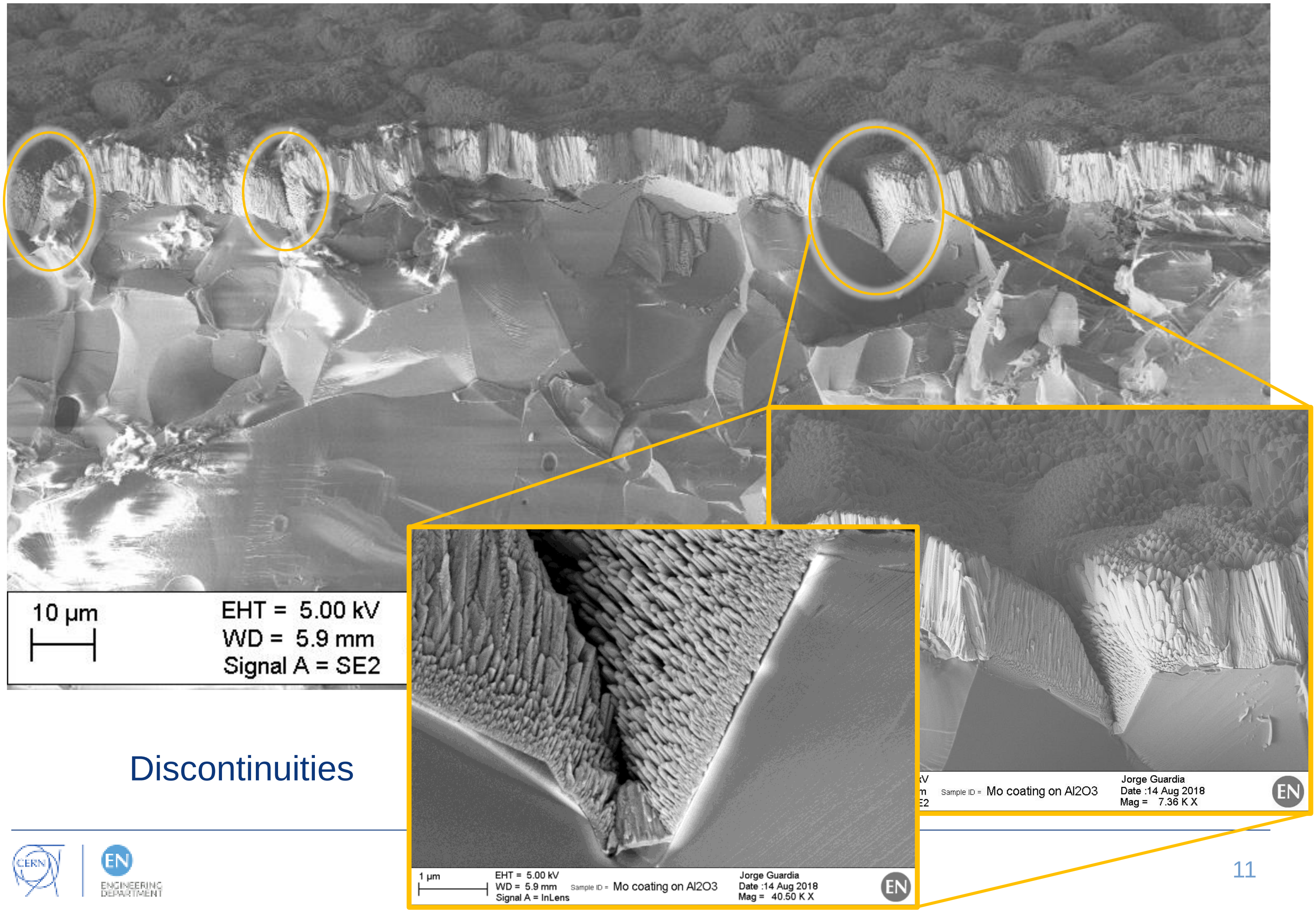
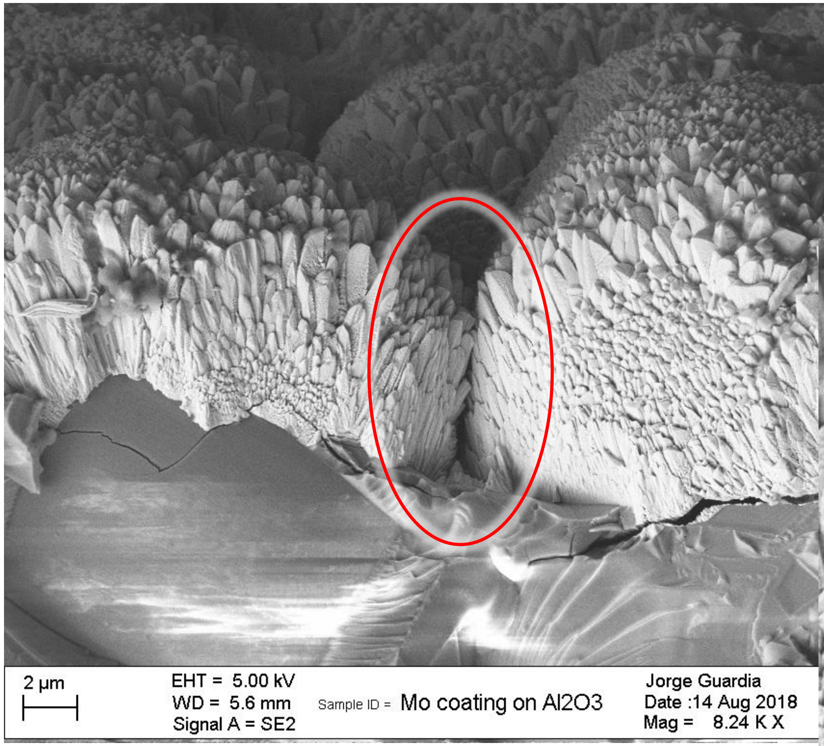
Mo on alumina

Mo on Alumina fracture surface

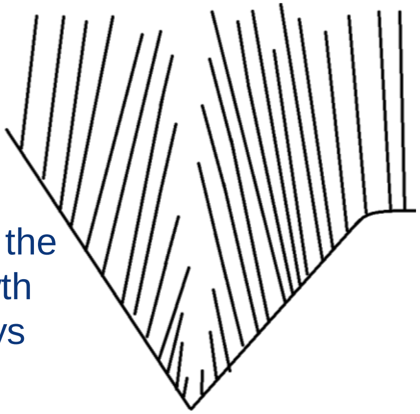


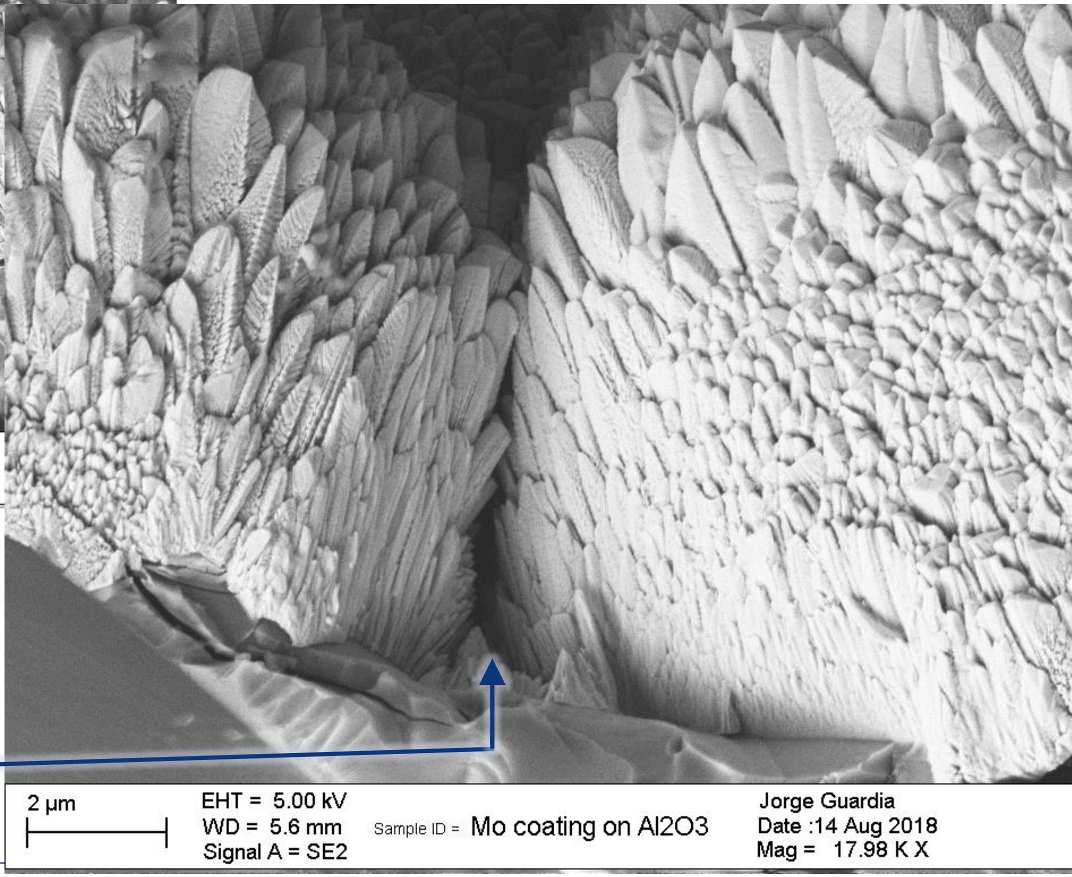
Discontinuities

Mo on Alumina fracture surface. Discontinuities



Schematic of the thin-film growth in deep valleys

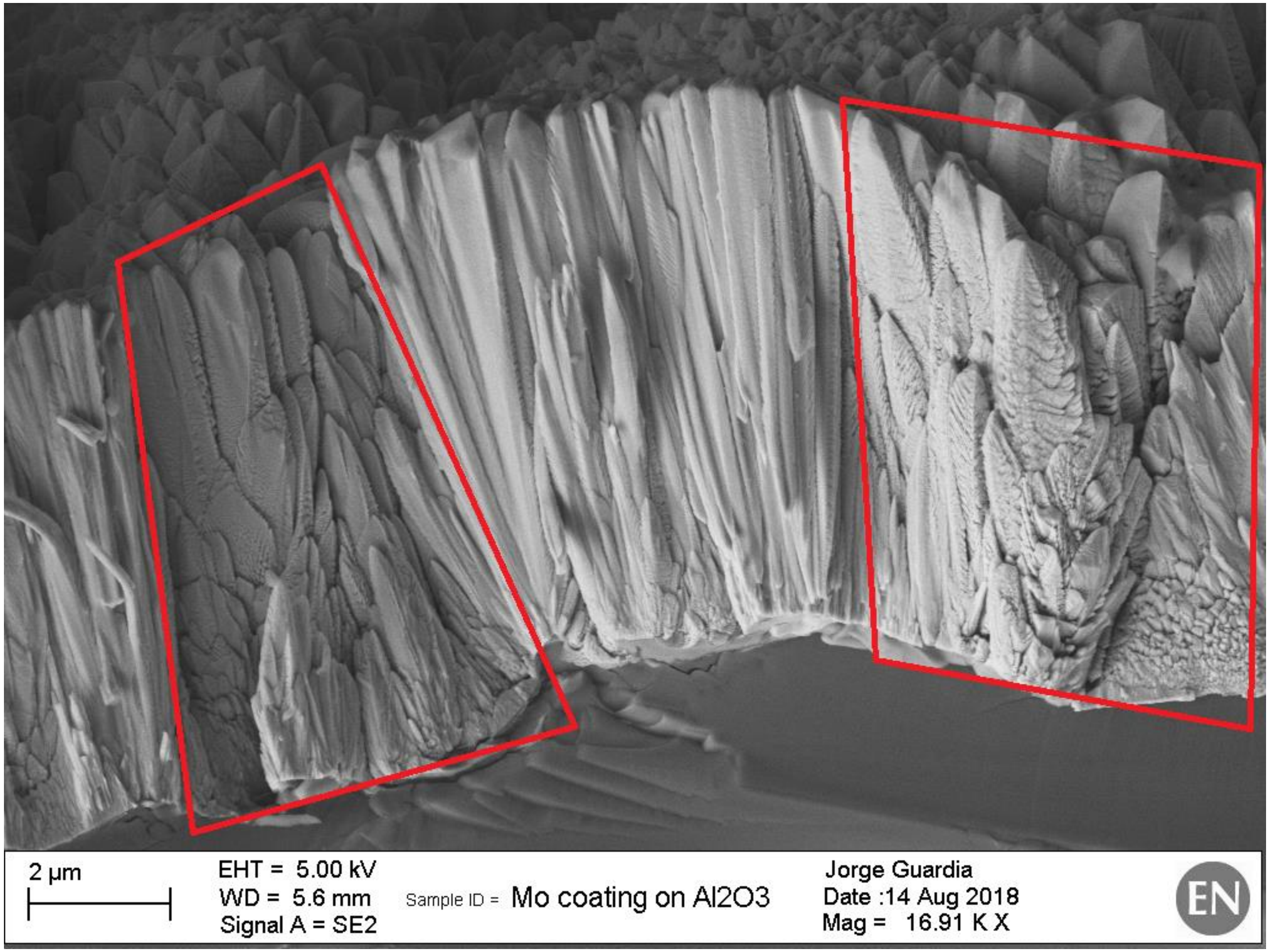




In deep valleys, the grains have irregular growing speed (shadowed deposition), forming a stair of grains with different heights that creates a discontinuity.

Bad contact

Mo on Alumina fracture surface

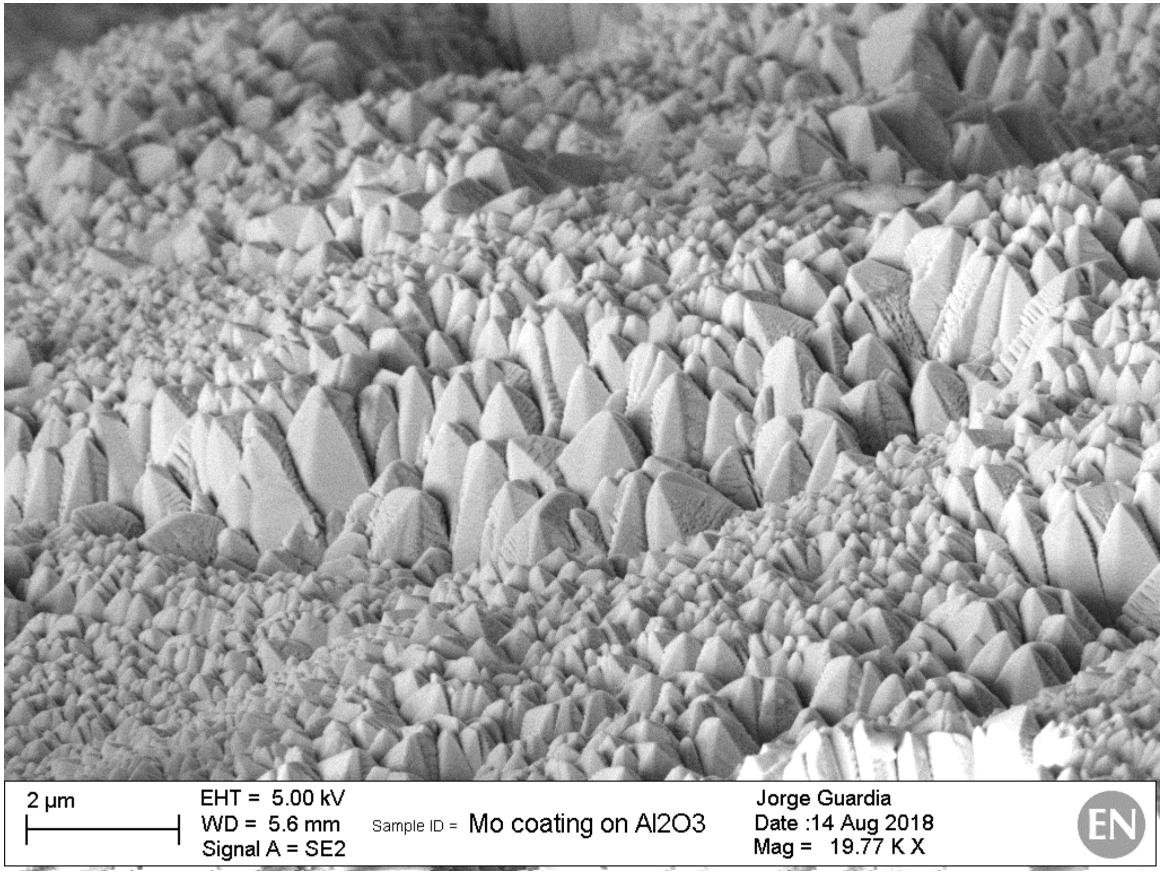


Areas with that kind of stairs of grains with different heights are visible in the fracture surface (in red).

This is because the discontinuity is a weak point and the fracture developed there (bad contact)

The other areas show a different morphology (intergranular fracture), showing that there was good cohesion there.

Mo on Alumina. Side view

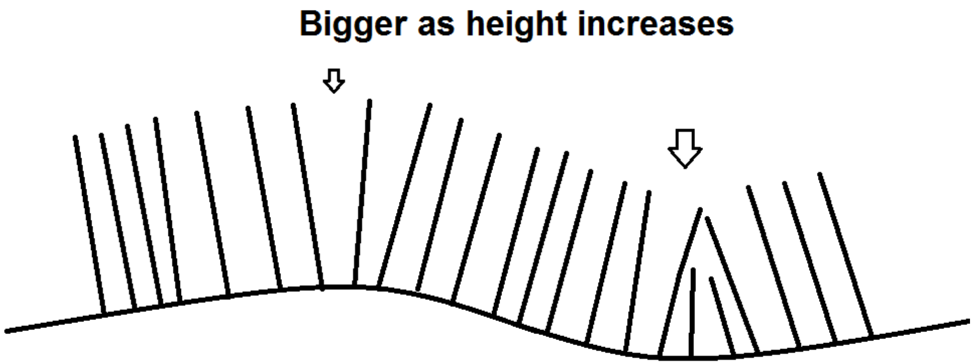


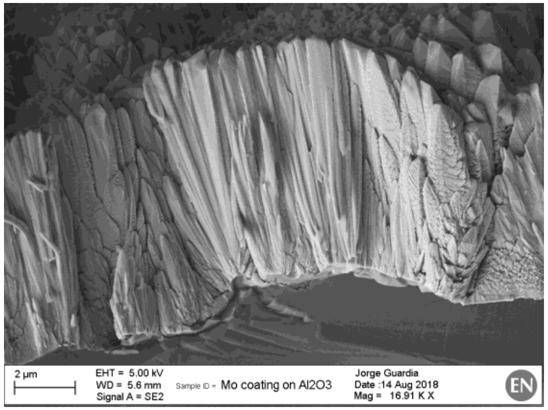
Grain (lateral) size at the surface is approximately between 0.3 and 2µm

Why are there smaller grains on glass?

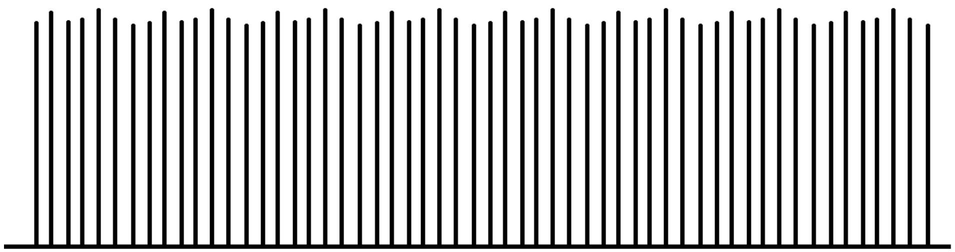
- Most likely, there is an effect of the roughness:

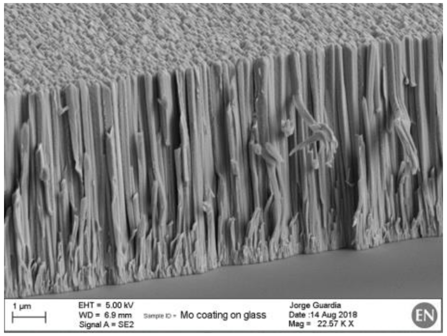
On substrates other than glass, the waves on the surface make some grains to grow more than others and block their growth, so the average grain size increases.





The coating on glass shows very homogeneous small grains because all are in the same geometrical conditions to grow.

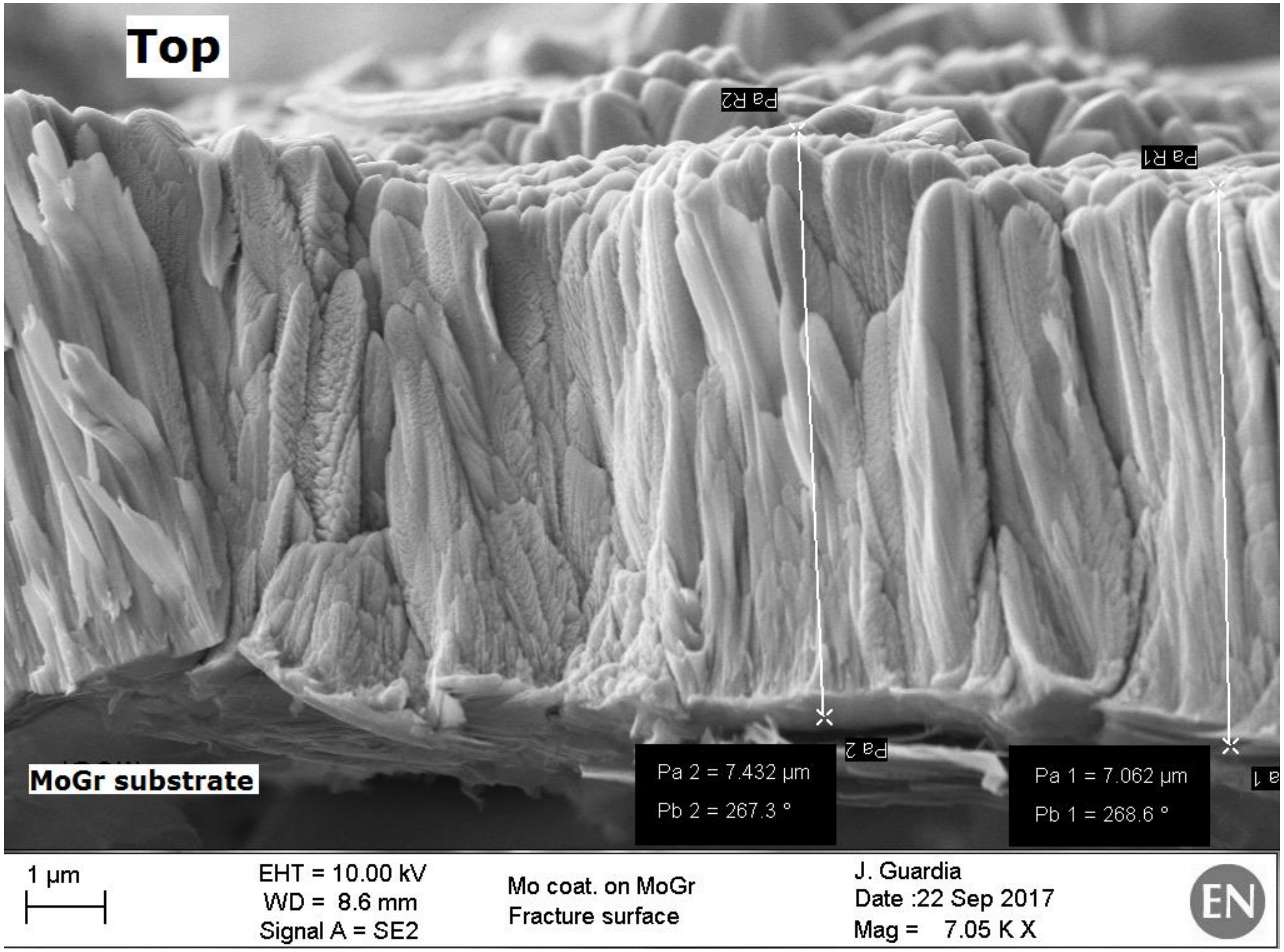




Mo on MoGr

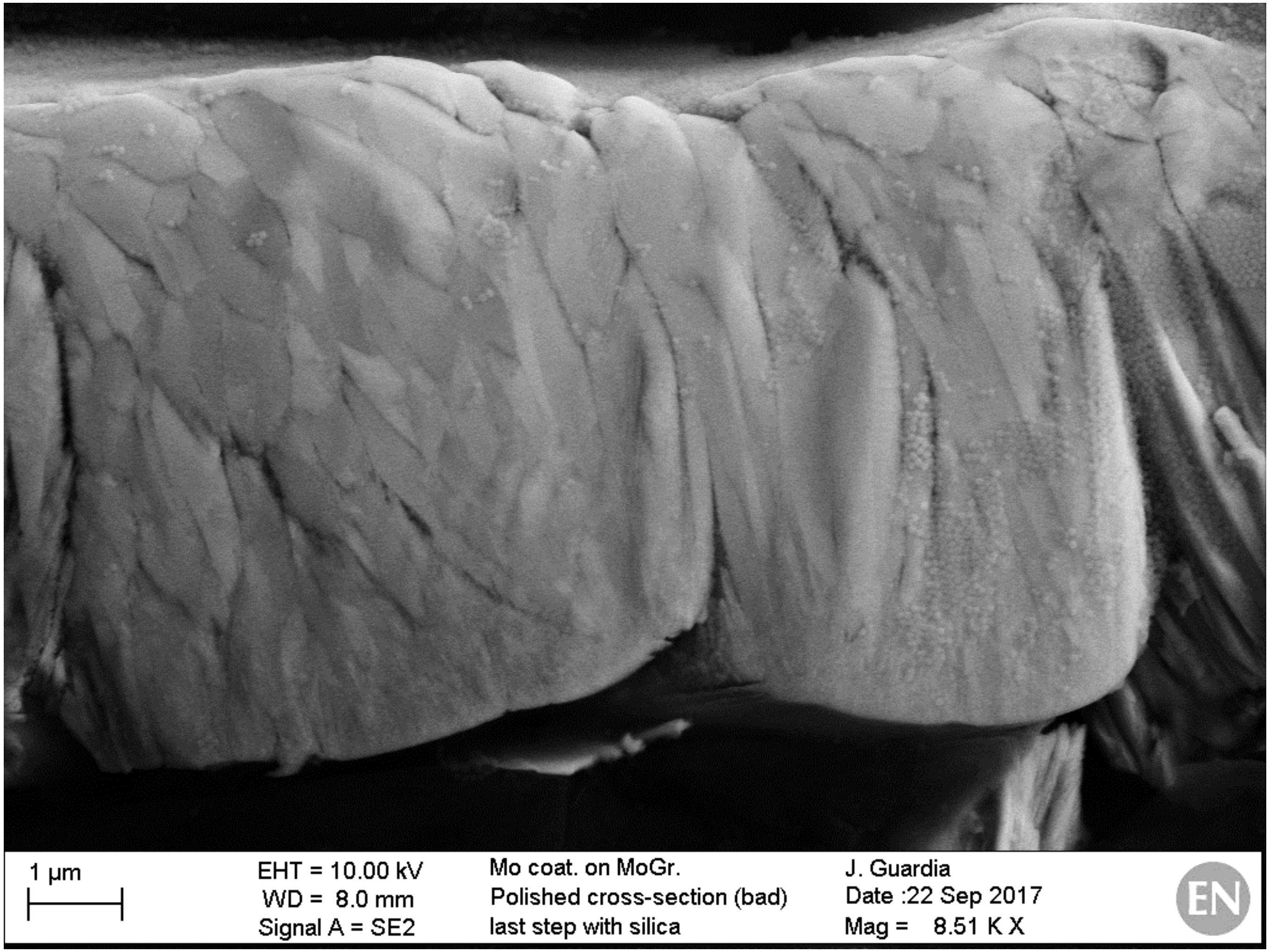
SEM of Mo coating on MoGr (Impedance meeting 20-4-2018)

https://indico.cern.ch/event/664498/



Grain (lateral) size close to the surface is approximately 0.5um

Mo on MoGr



SEM image showing contrast between different grains (polished surface, not very nice preparation). This allows clear identification of grain size and shape.

The spherical particles are silica, coming from the preparation of the sample.

Average grain size close to the top surface is approximately 0.5µm, slightly bigger than on glass (0.3µm)

Alumina-MoGr differences (discontinuities) explained by the roughness:

→ Roughness measurements of the substrates without coating

Roughness measurements (Impedance meeting 20-4-2018)

https://indico.cern.ch/event/664498/

Specimen	Surface treatment	Coating	Rq	Ra	Rt	Rz	Method X-spac
Alumina	-	-	4.1	2.9	41.6	26.7	O. 2.5µm
Alumina	-	Mo	3	2.2	38.8	23.3	O. 2.5µm
CFC C07	Mach+US	-	9.2	6.8	74.8	47.5	O. 2.5µm
CFC C07	Mach+US	Mo	9.2	7	69.1	45.2	O. 2.5µm
MoGr M03	Mach+US	-	1.1	0.7	16.4	9.1	O. 2.5µm
MoGr M03	Mach+US	Mo	0.8	0.5	8.5	5	O. 2.5µm
MoGr M04	Mach+US	-	1	0.8	11.7	6.6	O. 2.5µm
MoGr M04	Pol+US	Mo	1.1	0.7	22.3	11.6	O. 2.5µm
MG-6403Fc	Mach	-	1.9	1.5	12.0	8.8	C. ?
MG-6541Fc	Mach	-	1.8	1.4	11.0	8.9	C. ?
CFC AC150K	Mach	-	4.6	3.5	46.8	23.5	C. ?
Gr R4550	Mach	-	1.4	1.0	10.3	8.3	C. ?

Mach.=Machining Pol=polishing US=Ultrasonic cleaning
O=Optical (non-contact) C=standard contact measurement X-spac=acquisition spacing

O→EDMS.1966152
C→EDMS.1907137

MoGr grade in M03 and M04 specimens is NA-8304Gb

Mo-coating compendium report: EDMS 2012661

Conclusions

- The resistivity of the coating is affected by the combination of grain size and defects (discontinuities). This seems to explain the resistivity results

Nicolò's team measurements

	Substrate roughness	Mo grain size (average)	Amount of coating discontinuities	Coating conductivity (MS/m)		Coating resistivity (nΩ.m)
Glass	~0	+	no	+ 😐	4.3 [DC] 5.0 [RF]	232 [DC] 200 [RF]
Alumina	+++	++	++	+ 😐	4.6 [DC] 4.1 [RF]	218 [DC] 244 [RF]
MoGr	+	++	+	+++ ☺	- 14.3-16.7 [RF]	- 60-70 [RF]
CFC	++++	++	(big voids)	- ☹	n.d. (≈substrate)	n.d. (≈substrate)

MoGr #M04

- The discontinuities are created in the deep valleys (too rough substrate)
- Too flat substrate is not good either for low resistivity → smaller grains (<300nm) and low adherence
- More comprehensive studies of grain size can be performed if needed (polishing + SEM or FIB), more in background slides.
- Thermal treatments to increase grain size could be investigated, above Mo recrystallization temperature (900-1300°C [1]). Problems: coating detachment, Mo+C→carbide, gas influence during treatment [2].

[1] On the Recrystallization Behavior of Technically Pure Molybdenum, S. Primig et al. 17th Plansee Seminar 2009, Vol. 1 https://www-plansee-com.azureedge.net/fileadmin/user_upload/On_the_Recrystallization_Behavior_of_Technically_Pure_Molybdenum_2009.pdf
[2] Effect of inert gases on the recrystallization of tungsten Yu M. Aleksandrova et al. Fiziko-Khimicheskaya Mekhanika Materialov, Vol 2, No 3, pp. 327-332, 1966. https://link.springer.com/content/pdf/10.1007%2FBF00714677.pdf

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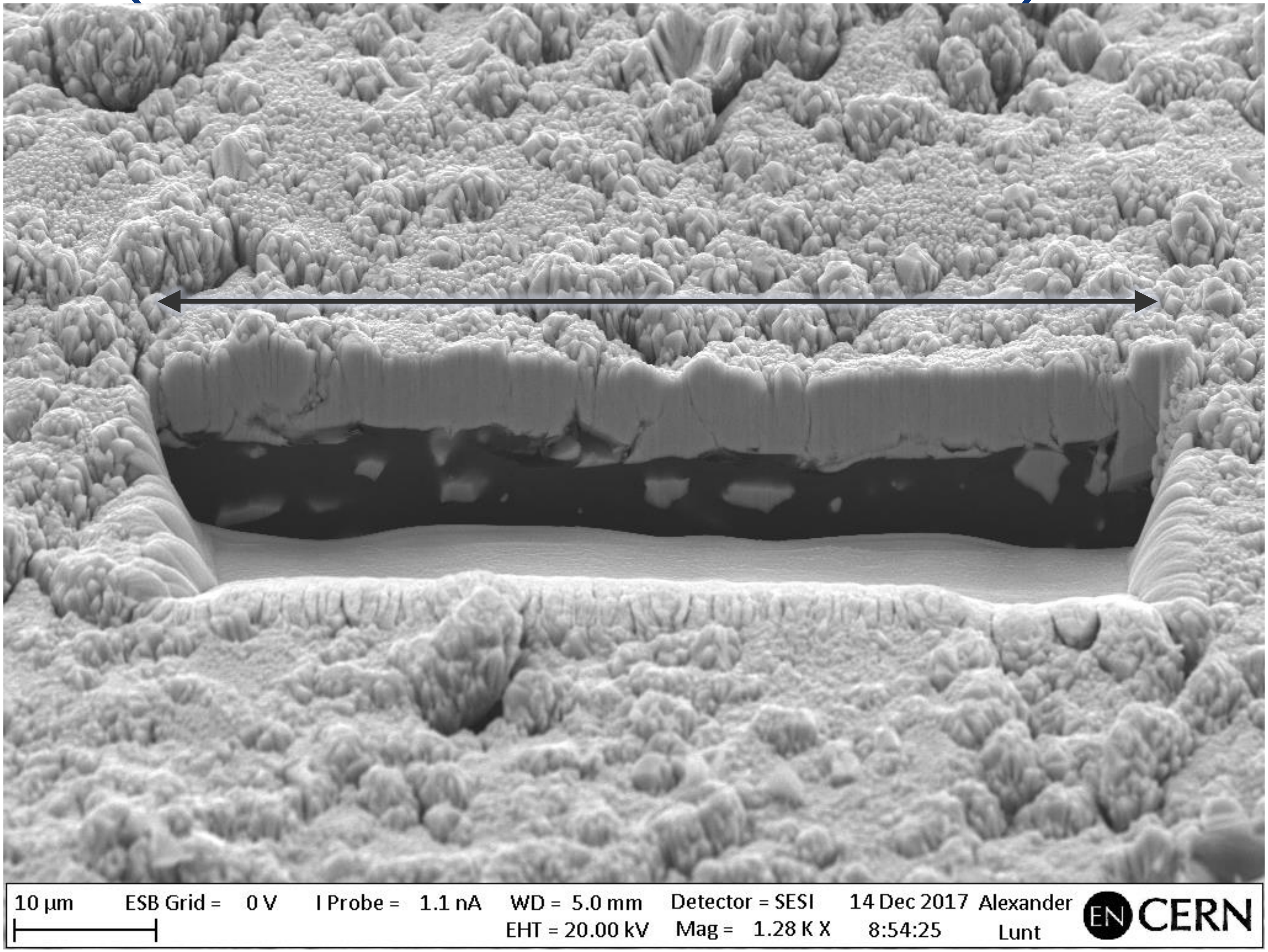
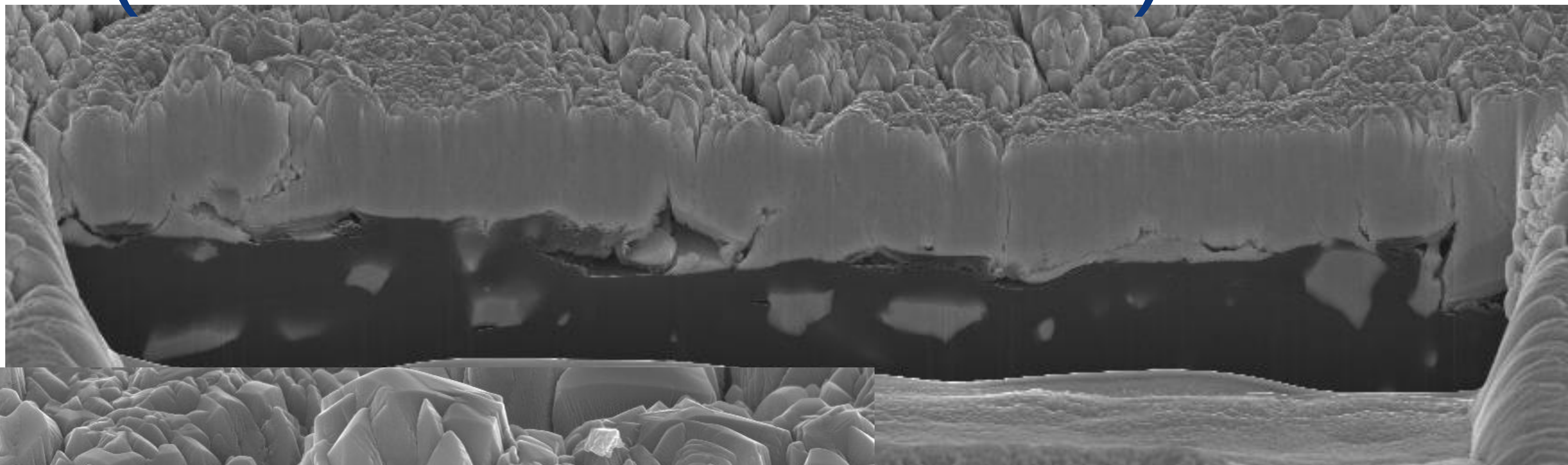
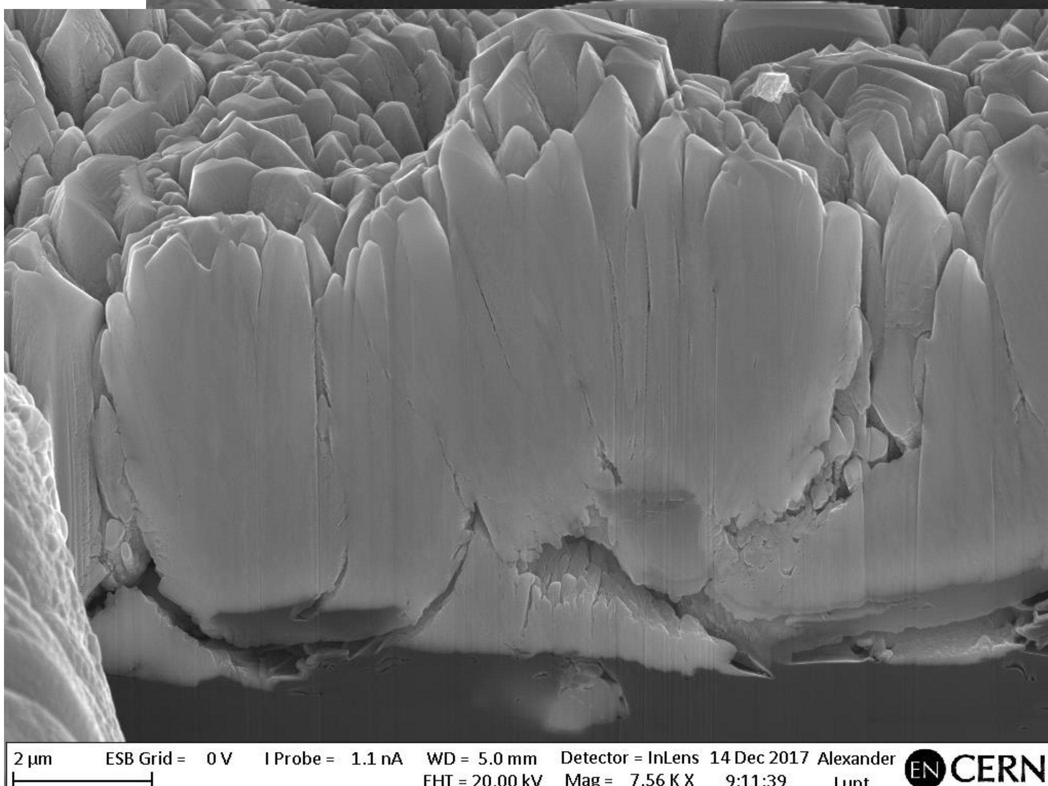
FIB (Mo on MoGr NB-8304Je)

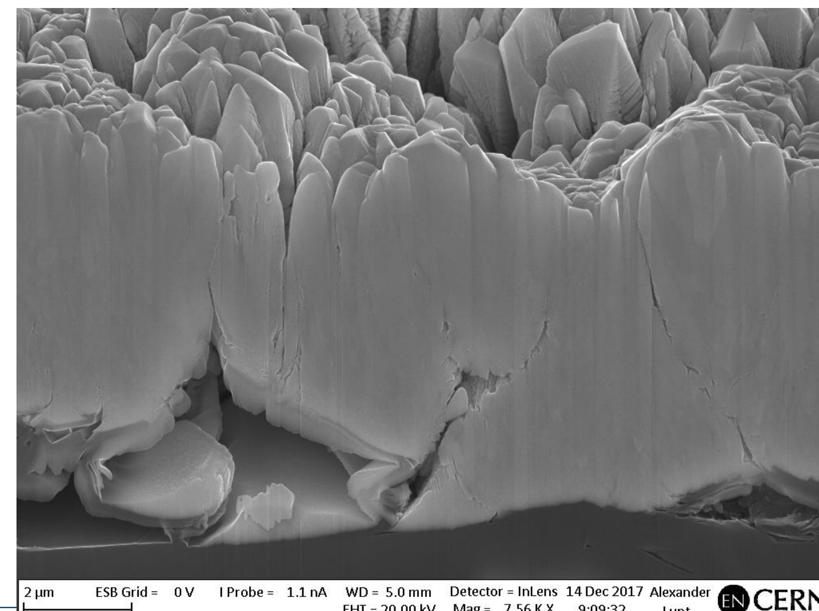


local study:
~60µm

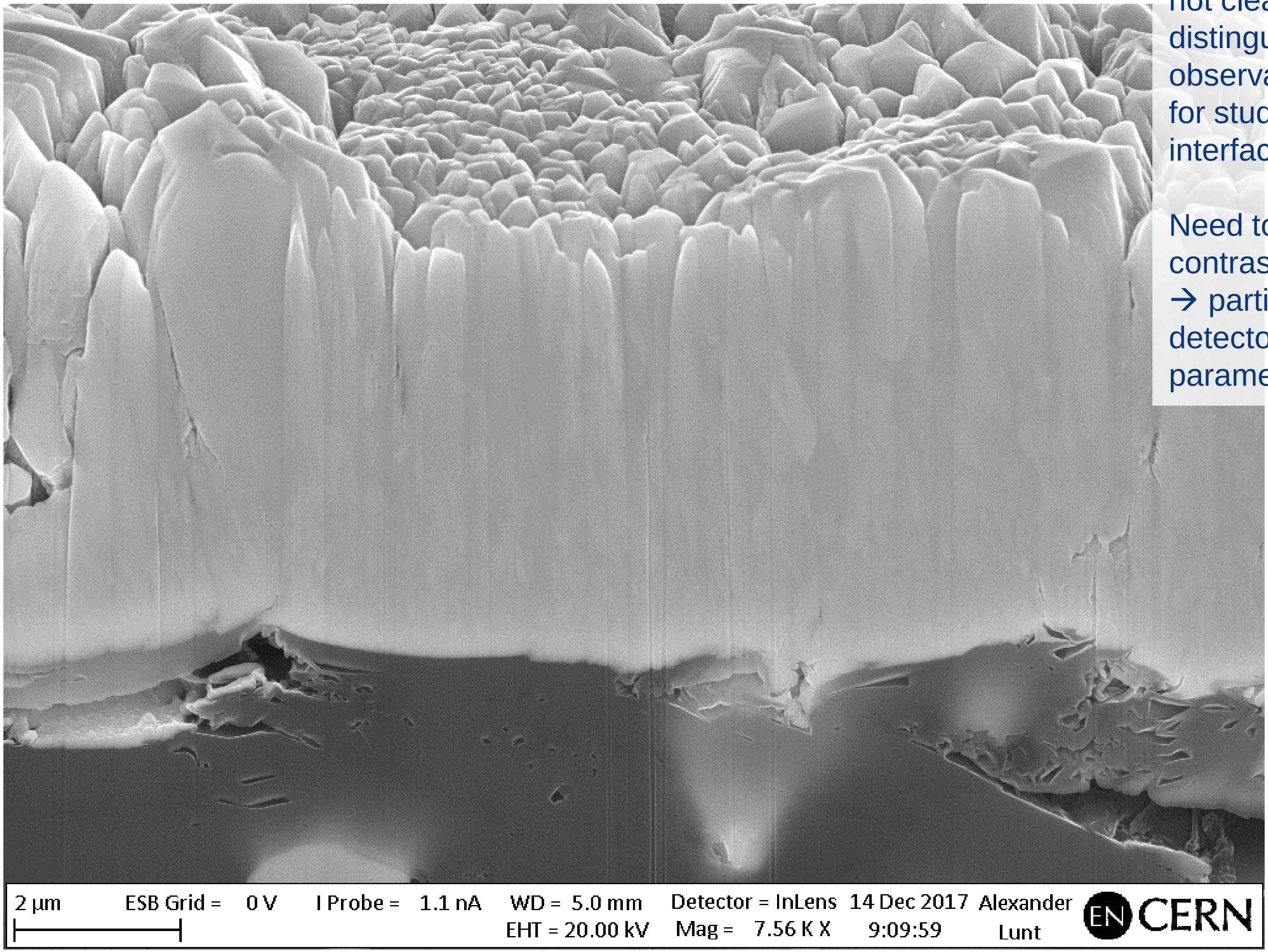
FIB (Mo on MoGr NB-8304Je)







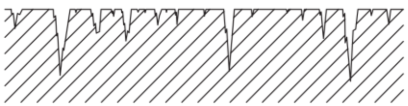
FIB (Mo on MoGr NB-8304Je)



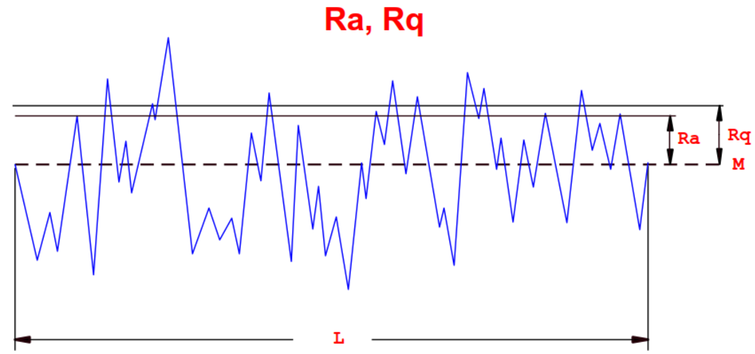
In this case, grains are not clearly distinguishable (these observations were done for studying the interface)

Need to improve contrast between grains → particular SEM detector/ beam parameters

Roughness definitions

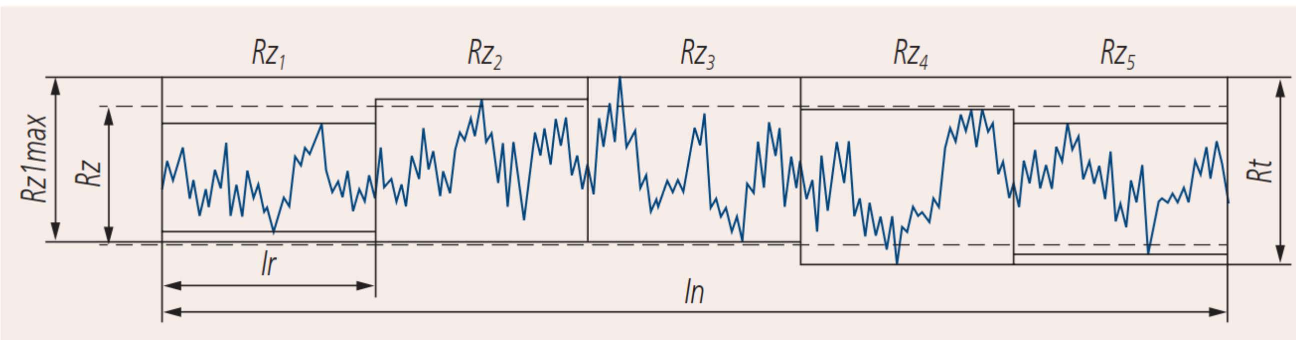
Surface Profile	R_a
	0.2
	0.2

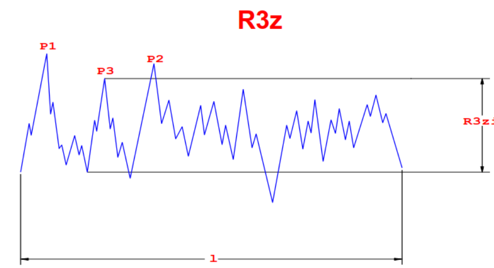
Even with identical Ra values, the performance of the surface may be very different



Roughness Average, *Ra*, is the arithmetic average of the absolute values of the profile heights over the evaluation length.

RMS Roughness, *Rq*, is the root mean square average of the profile heights over the evaluation length





Third Maximum Peak-to-Valley Height, *R3z*, is the mean of the third maximum peak-to-valley heights in the evaluation length.

Rt – total height of the roughness profile: Difference between height **Zp** of the highest peak and depth **Zv** of the deepest valley within the evaluation length ln (Figure 7).

Rz_i – greatest height of the roughness profile: Sum of the height of the highest profile peak and the depth of the deepest profile valley, relative to the mean line, within a sampling length **lr_i**.

Rz1max – maximum roughness depth: Largest of the five **Rz_i** values from the five sampling lengths lri within the evaluation length ln.

Rz – mean roughness depth: Mean value of the five Rzi values from the five sampling lengths lr_i within the evaluation length ln.

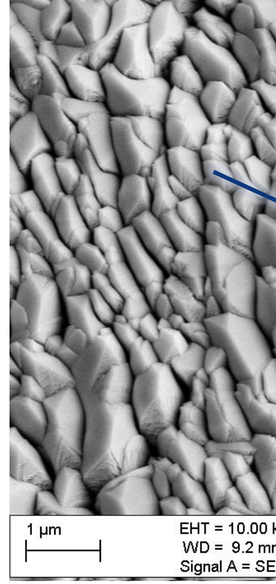
Refs: Mituyoyo Quick guide to roughness measurement Bulletin No. 2229 (2016)
https://www.mitutoyo.com/wp-content/uploads/2012/11/1984_Surf_Roughness_PG.pdf

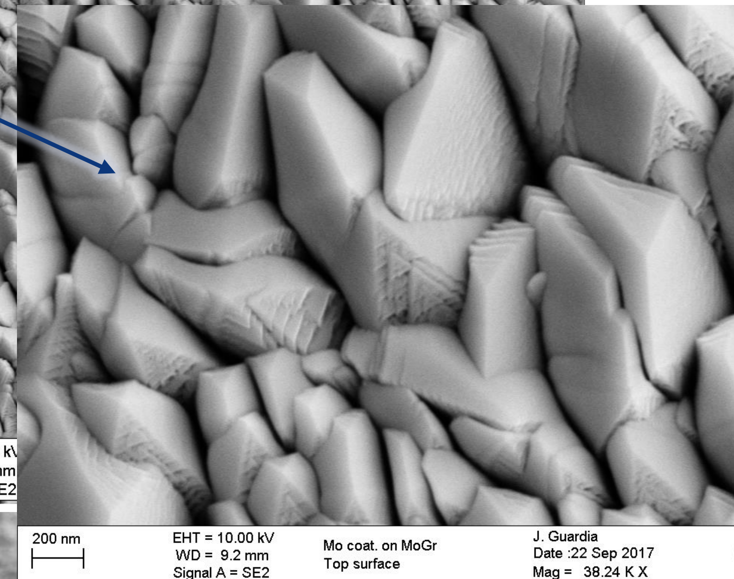
http://www.predev.com/pdffiles/surface_roughness_terminology_and_parameters.pdf
https://www.tss-static.com/remotemedia/media/globalformastercontent/products/staticseals/airseal/files/aerospace_gb.pdf

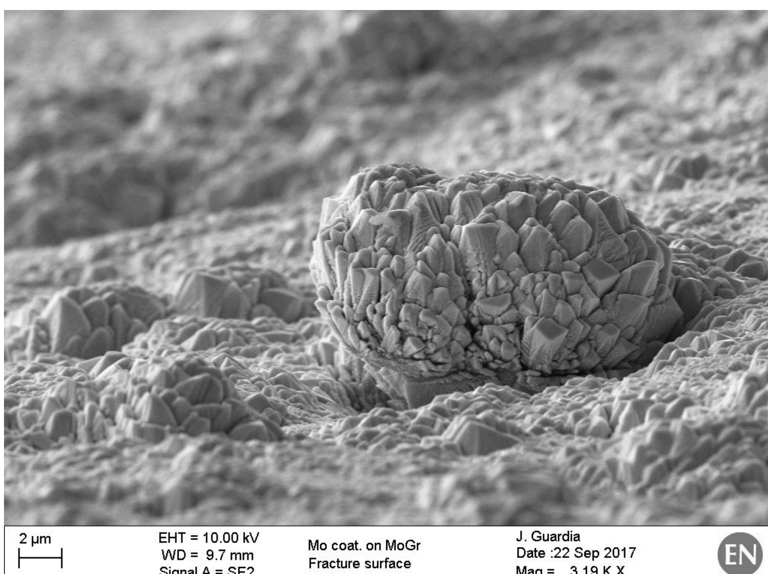
Observations shown in past meetings

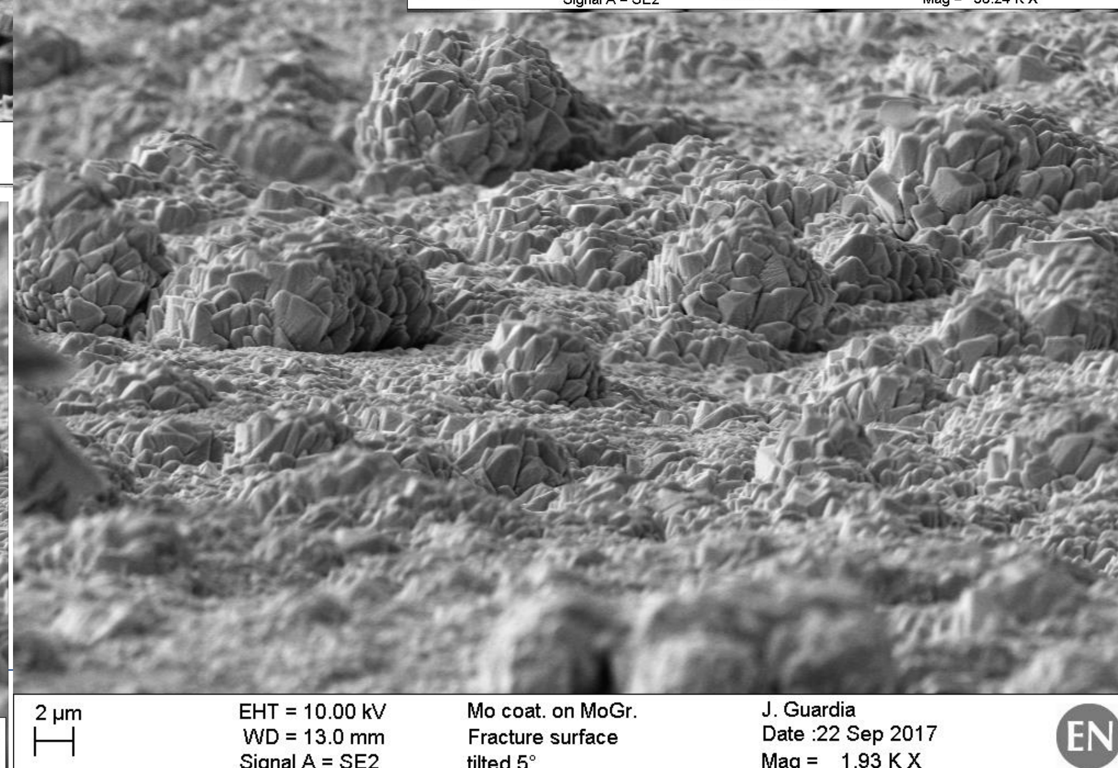
Mo on MoGr


MoGr
20 µm
EHT = 10.00 kV
WD = 12.1 mm
Signal A = SE2
Mo coat. on MoGr.
Fracture surface
J. Guardia
Date :22 Sep 2017
Mag = 324 X

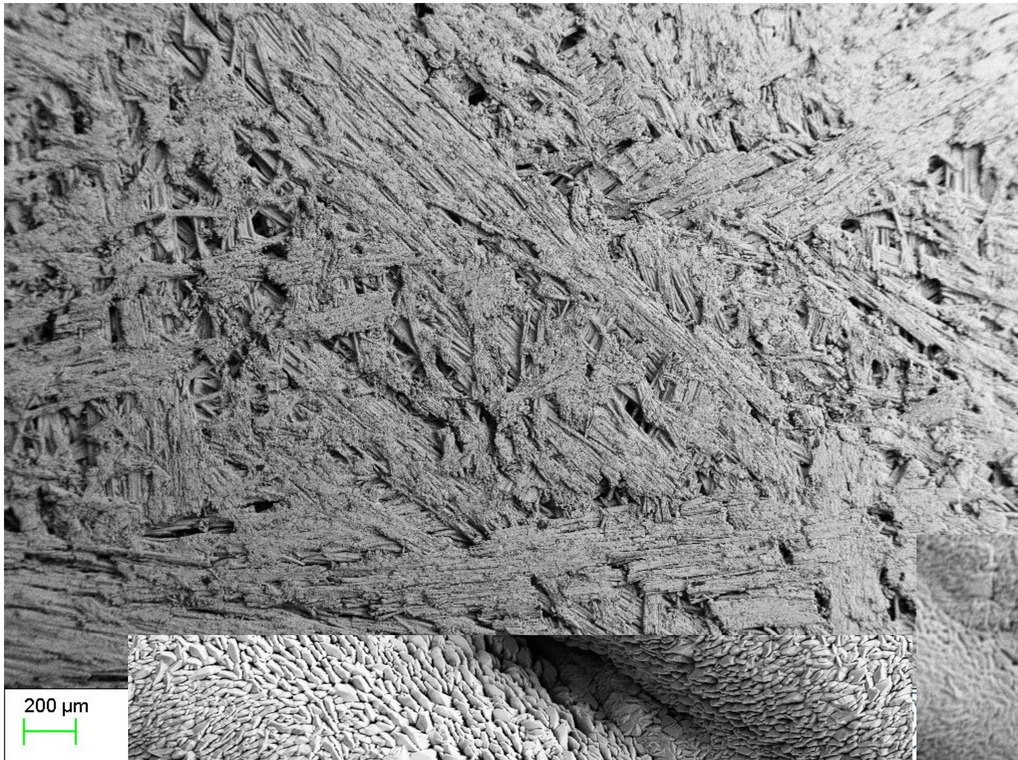

1 µm
EHT = 10.00 kV
WD = 9.2 mm
Signal A = SE2

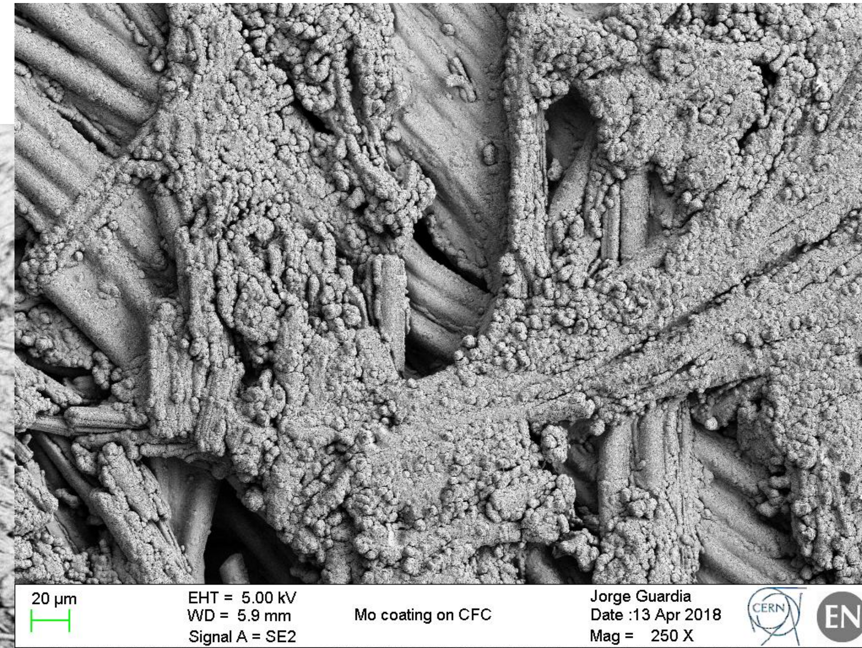

200 nm
EHT = 10.00 kV
WD = 9.2 mm
Signal A = SE2
Mo coat. on MoGr
Top surface
J. Guardia
Date :22 Sep 2017
Mag = 38.24 K X

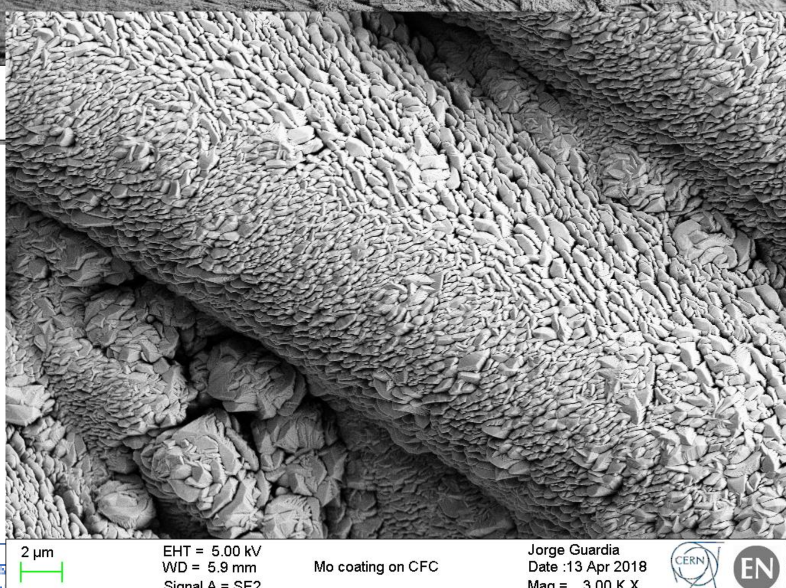

2 µm
EHT = 10.00 kV
WD = 9.7 mm
Signal A = SE2
Mo coat. on MoGr
Fracture surface
J. Guardia
Date :22 Sep 2017
Mag = 3.19 K X

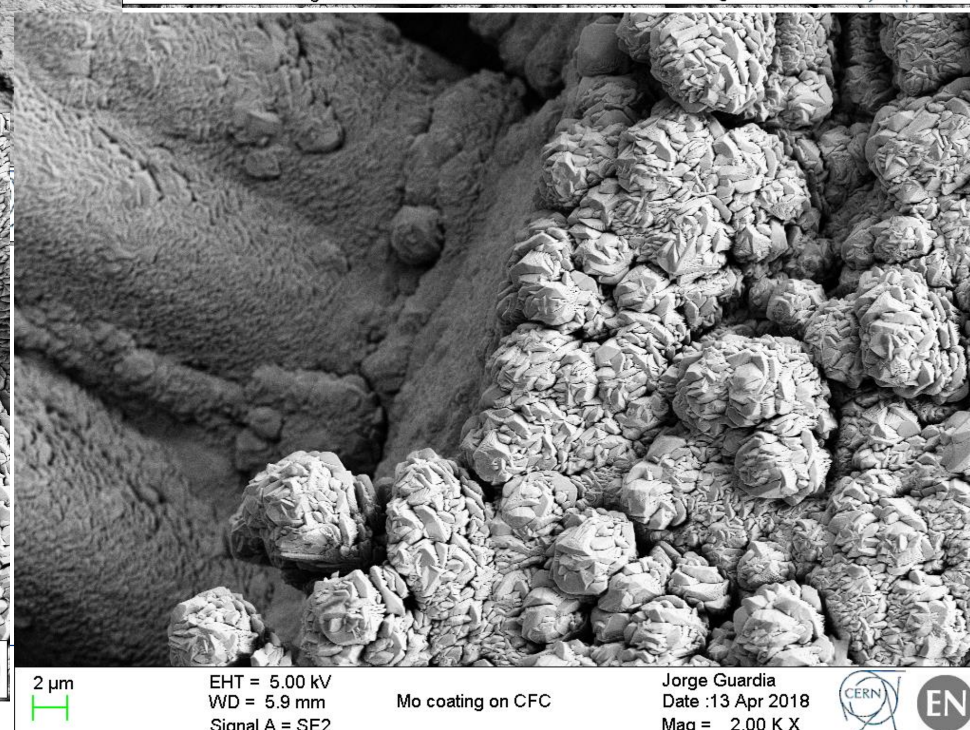

2 µm
EHT = 10.00 kV
WD = 13.0 mm
Signal A = SE2
Mo coat. on MoGr.
Fracture surface
tilted 5°
J. Guardia
Date :22 Sep 2017
Mag = 1.93 K X

Mo on CFC FS140

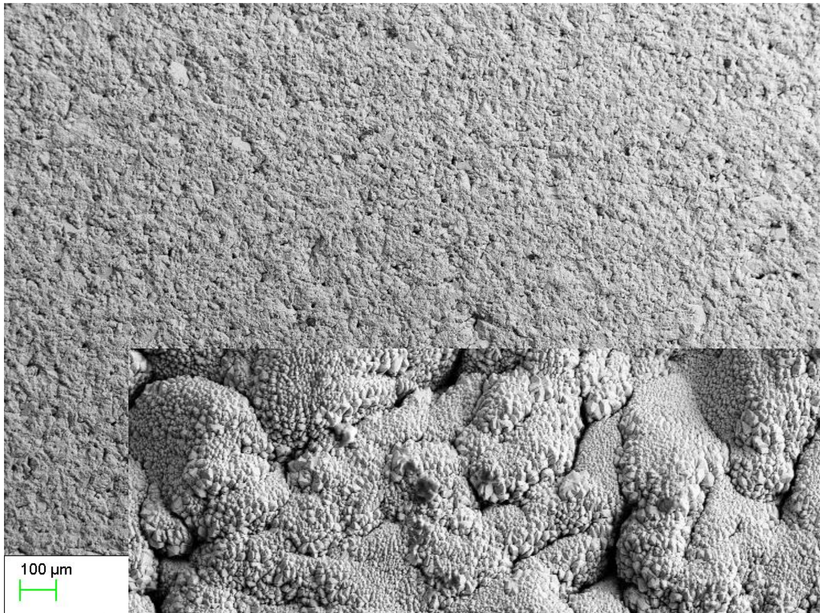


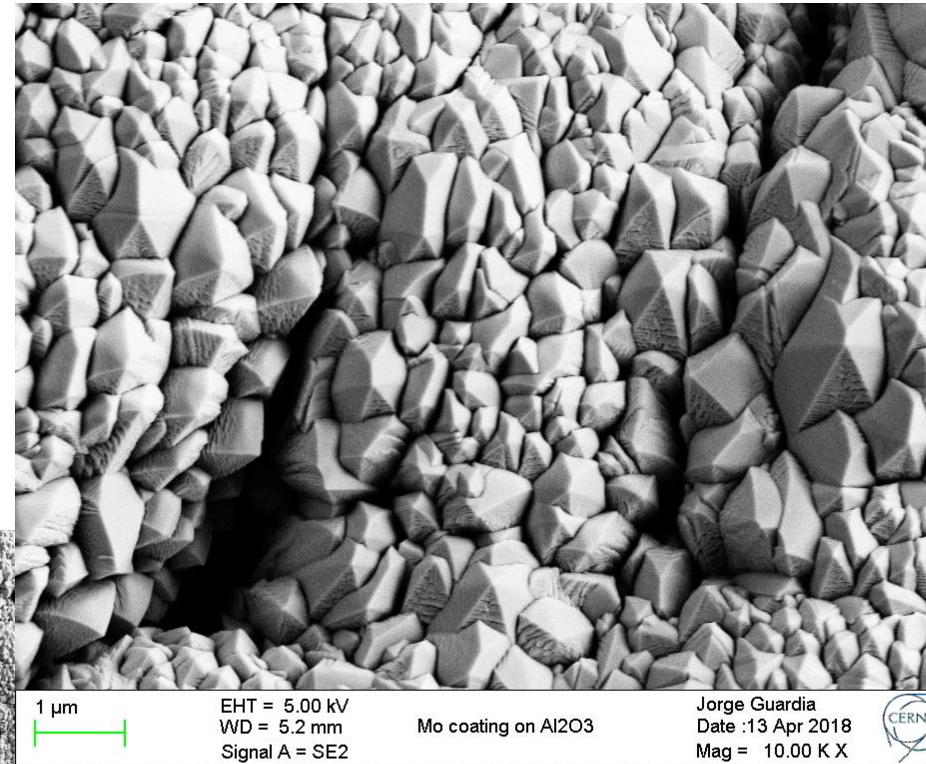


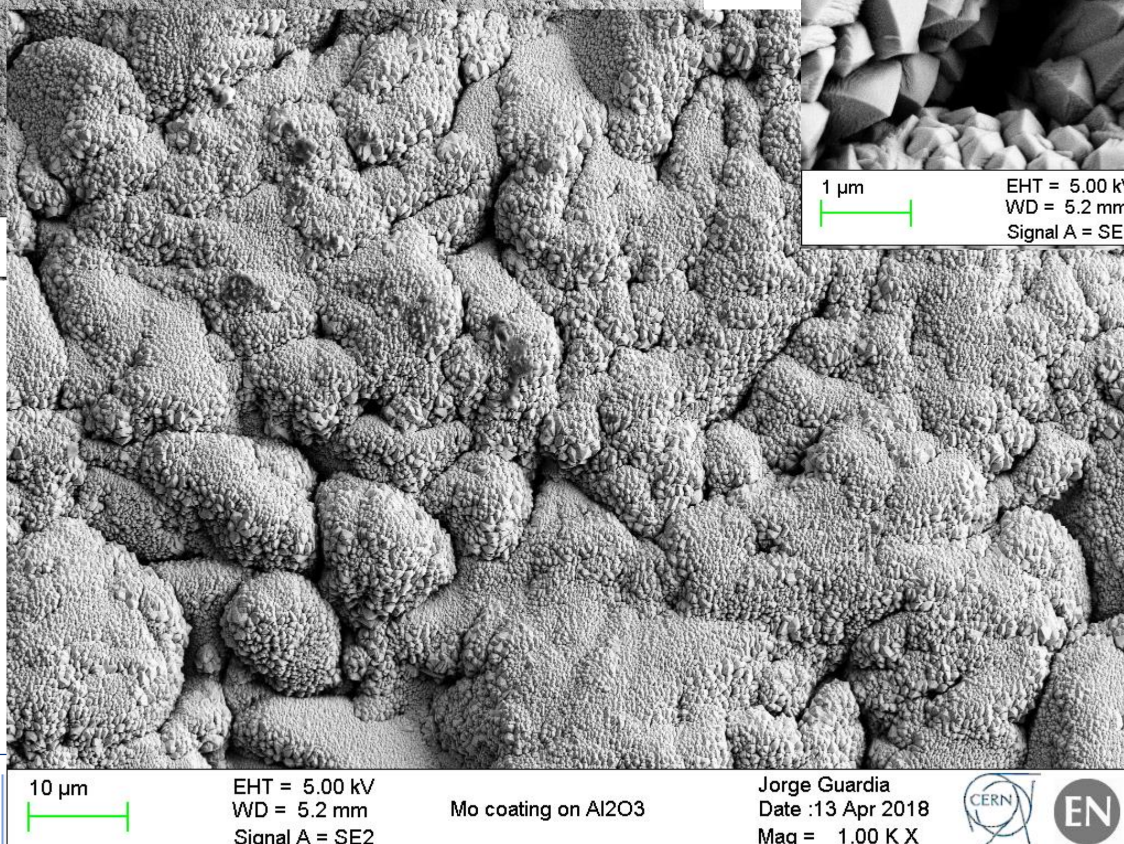




Mo on Al_2O_3


100 µm


1 µm
EHT = 5.00 kV
WD = 5.2 mm
Signal A = SE2
Mo coating on Al2O3
Jorge Guardia
Date :13 Apr 2018
Mag = 10.00 K X


10 µm
EHT = 5.00 kV
WD = 5.2 mm
Signal A = SE2
Mo coating on Al2O3
Jorge Guardia
Date :13 Apr 2018
Mag = 1.00 K X