



*Microstructural characterization of mechanical
and corrosion wear mechanisms of bio-tribological
coatings, carried out by TEM technique*

www.imim.pl

Łukasz Major

Project WND-POWR.03.02.00-00-1043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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Suggestions for topics

- 1). Thin Films Deposition-
- 2). Nucleation and Growth of Thin Films-
- 3). Materials Design Inspired by Nature-
- 4). Sample Preparation Techniques for Transmission Electron Microscopy-.....
- 5). The Transmission Electron Microscope (instrument, contrast mechanism, scanning transmission electron microscopy)
- 6). Structure Defects-
- 7). Standard Micromechanical and Tribological Testing-
- 8). In-situ Electron Microscopy-
- 9). Wear-
- 10). Corrosion-
- 11). Cell-Material Interaction and Biocompatibility-



Accredited Testing Laboratories

at the Institute of Metallurgy and Materials Science
of the Polish Academy of Sciences

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phone: (0048)(12) 295 28 00; e-mail: zlb@imim-pan.krakow.pl



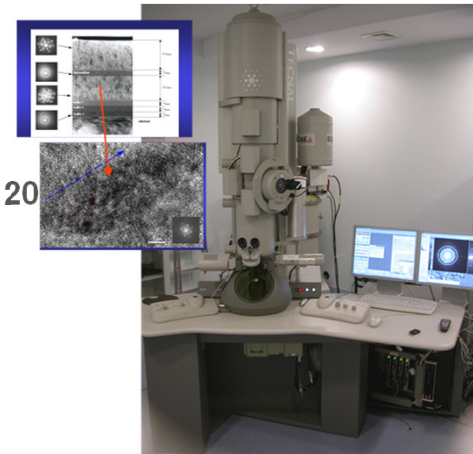
AB 120

LABORATORY OF ANALITICAL ELECTRON MICROSCOPY (L-2)

Authorisation:

Accredited activity is defined in the Scope of Accreditation No. AB 120

Quantitative and qualitative analysis of elements in alloys and ceramic materials,
identification of phases, analysis of chemical composition and shape of precipitates,
examination of structure of grain boundaries,
High resolution analysis (atomic scale)
determination of surface morphology, shape and size of inclusions.

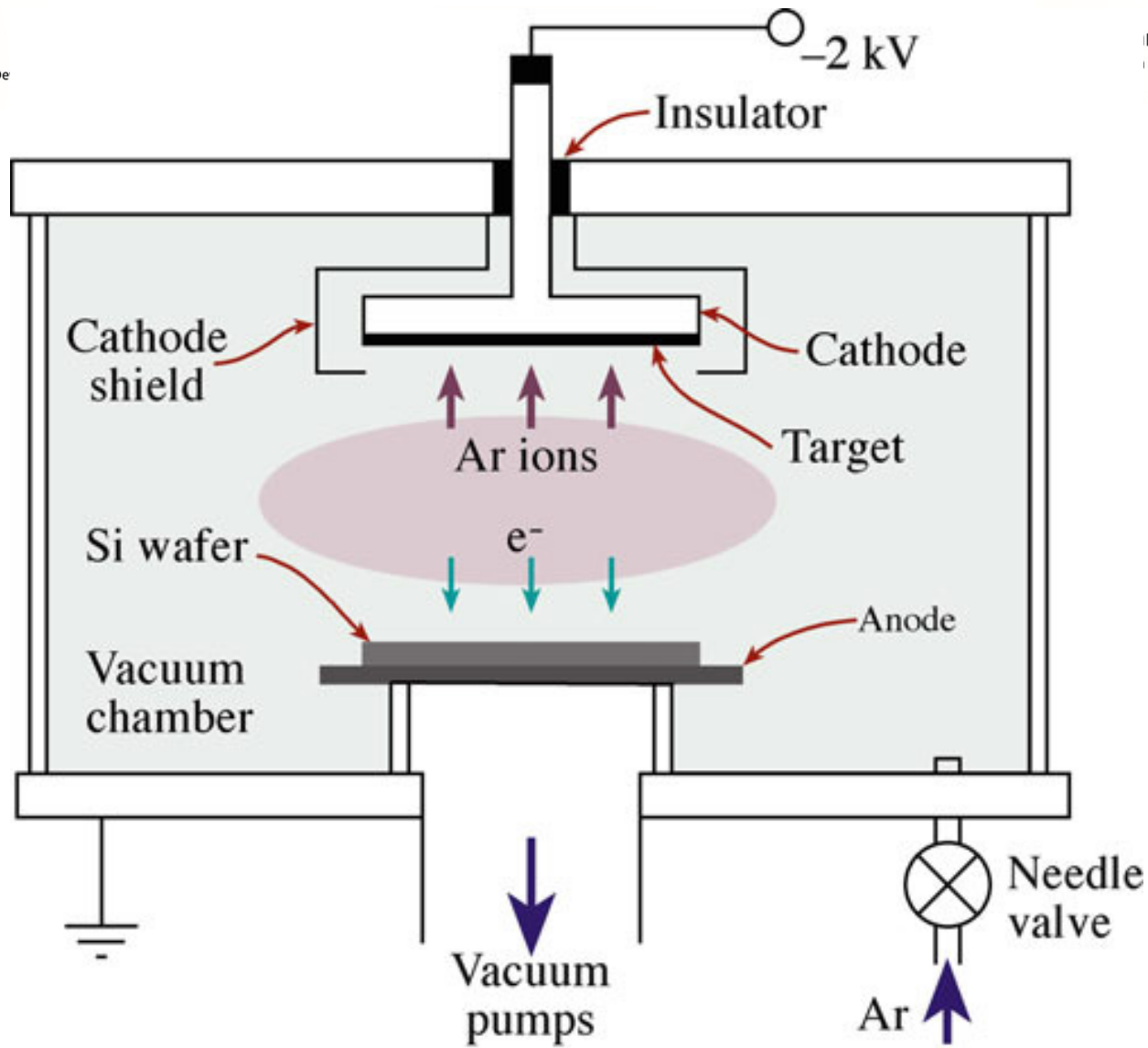


Institute of Metallurgy and Materials Science Polish Academy of Sciences

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High-vacuum deposition processes

Available online at www.sciencedirect.com
SCIENCE @ DIRECT®
Surface & Coatings Technology 200 (2005) 1439–1444
www.elsevier.com/locate/surftec

Industrially-scaled large-area and high-rate tribological coating by Pulsed Laser Deposition
Jürgen M. Lackner*
Laser Center Leoben, JOANNEUM RESEARCH Forschungsgesellschaft mbH, Leoben Strasse 94, A-8712 Niklasdorf, Austria
Available online 9 September 2005



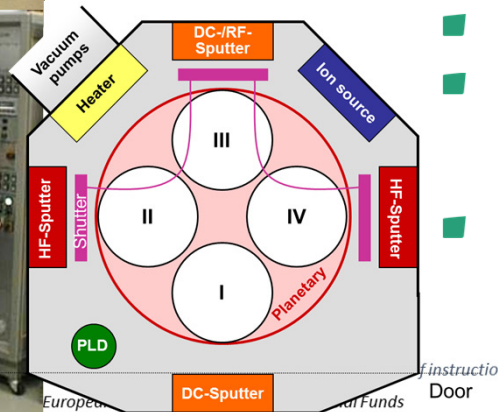
Pulsed Laser Deposition (PLD)

Magnetron sputtering (MS)

Anode Layer Ion Source treatment and assisted deposition (ALS)

- PLD: KrF & Nd:YAG multi-beam evaporator
- MS: DC, DC-pulsed, RF on 1-4 sputter magnetrons

- ALS: Veeco ALS 340
- Usable chamber volume: planetary diameter 560 mm, 450 mm
- Gas pressure: 10^{-4} to 10^{-2} mbar





Surgical tools/



Industrial partner



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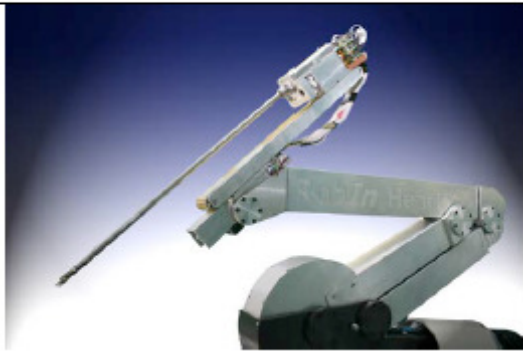
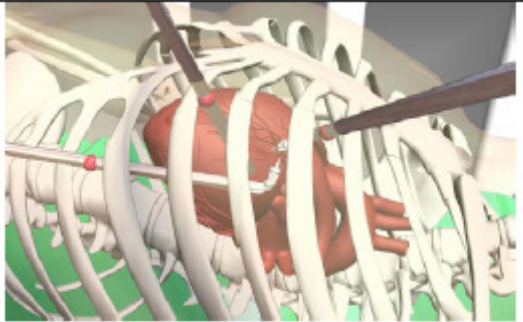
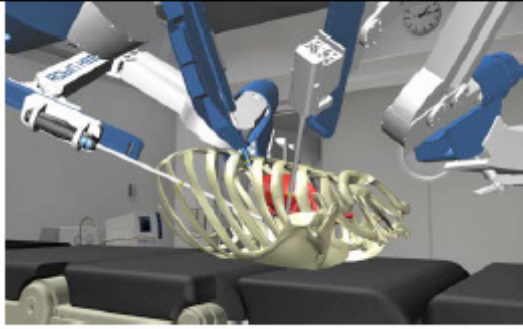
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ROBIN HEART



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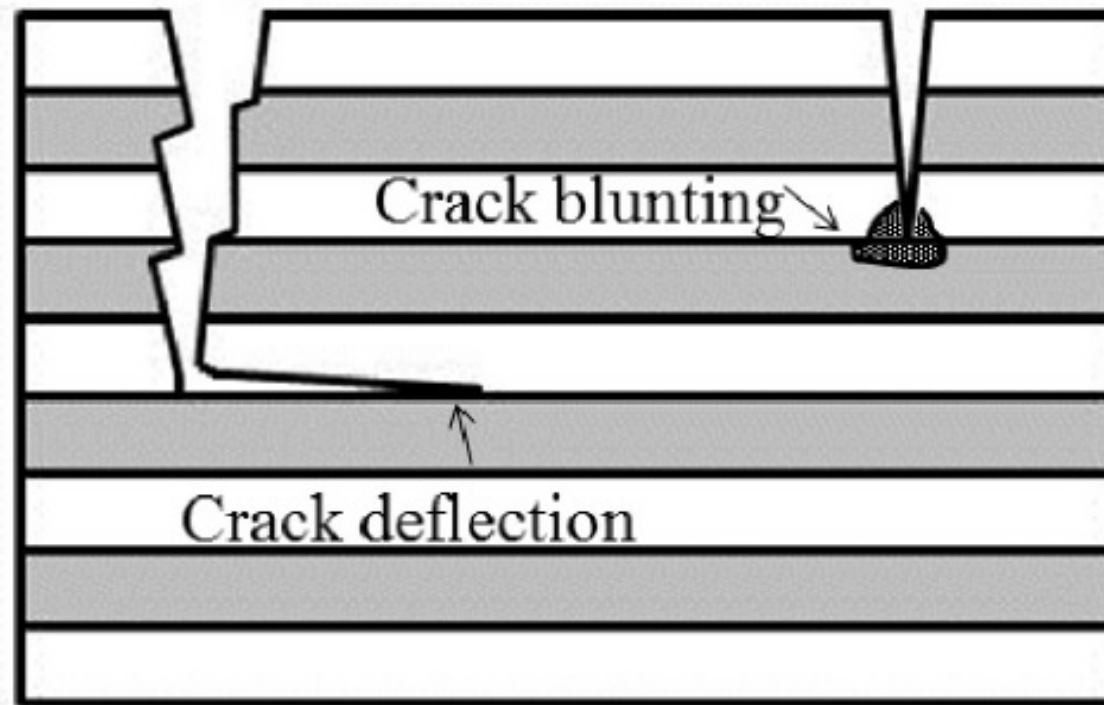


struction

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The role of the multilayer coating



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Biomimetics

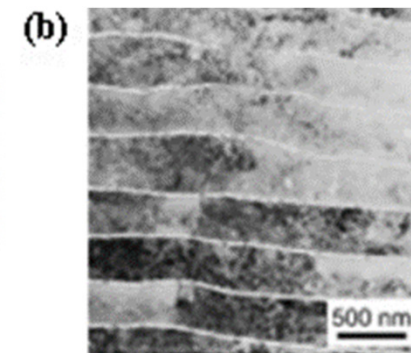
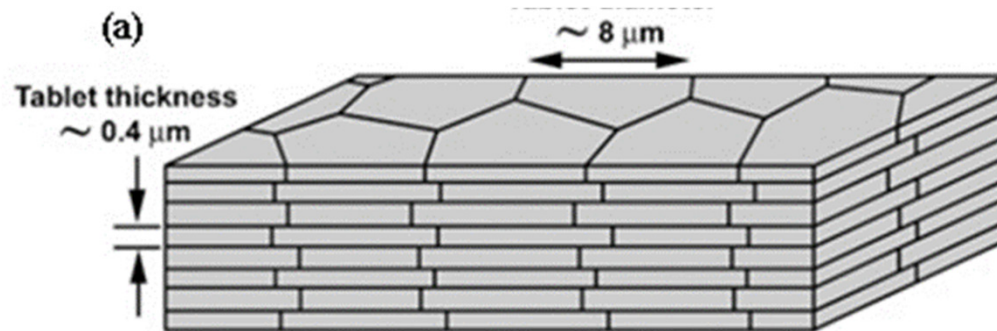
Biomimetics



Study and simulation of biological systems with desired properties

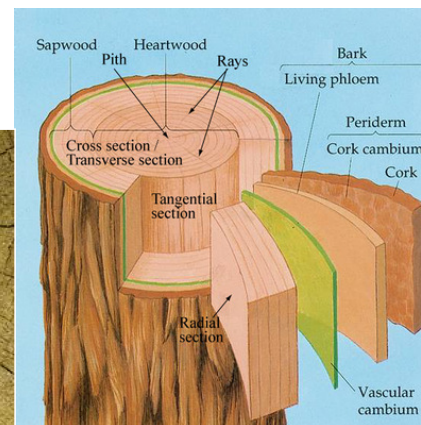
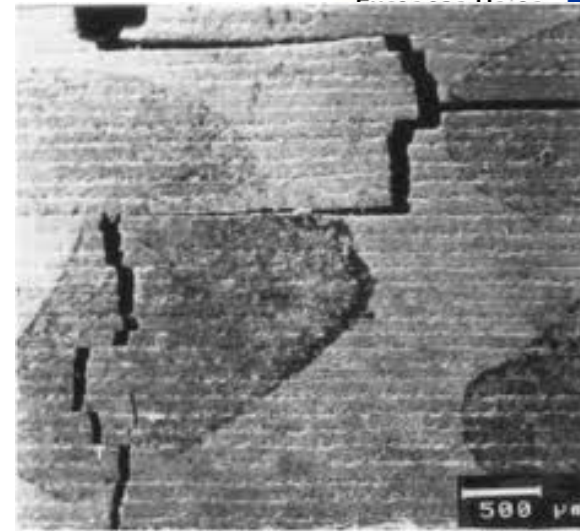
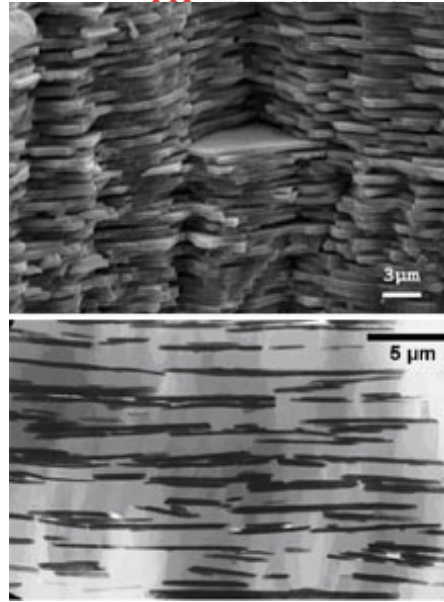


Transformation of the underlying principles into man-made technology





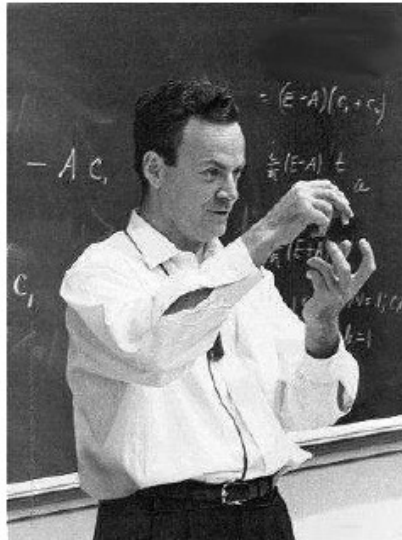
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In 1959. **Richard Feynman gives his famed** **talk “There is Plenty of Room at the Bottom”**



Richard Feynman © 1965

“What I want to talk about is the problem of manipulating and controlling things on a small scale.”

In this talk, Feynman said that we have progressed to the point where we can and should manipulate matter at what today we call the nano-scale.

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Resolution



Ability to distinguish between 2 closely spaced points.



Resolution of human eye:
100 μm
(100 μm = 0.0039 inches)

If $> 100 \mu\text{m}$



Will see 2
objects

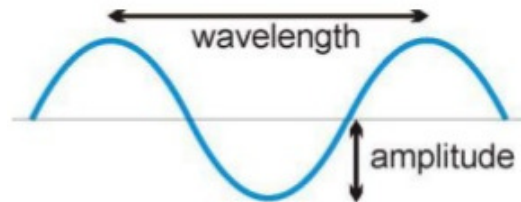
If $< 100 \mu\text{m}$



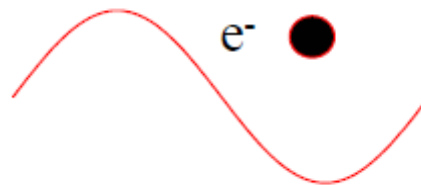
Will see only
1 object



In order to see the element in the size of „d” it is a need to have $\lambda < d$. For example the length of visible light is $\rightarrow 0.5 \mu m$



In general, to receive information about atomic structure it is necessary to apply $\lambda < 0.1 \text{ nm}$

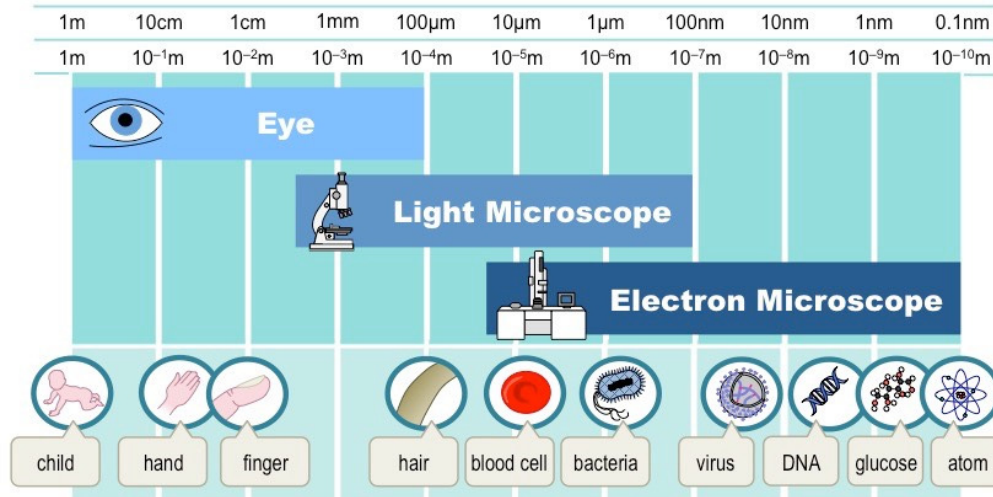


$\lambda = \sqrt{150/V}$, where
 λ - wavelength in Angstroms,
V- accelerating voltage in volts

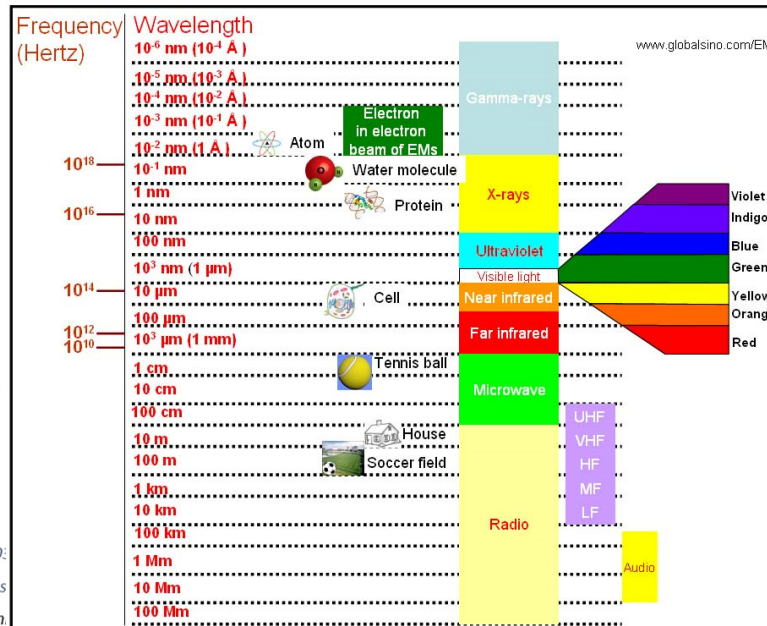
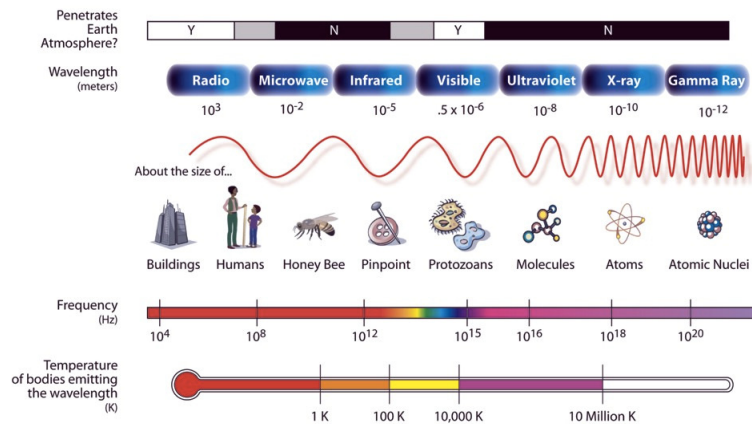
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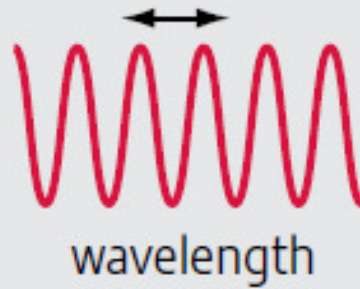


THE ELECTROMAGNETIC SPECTRUM

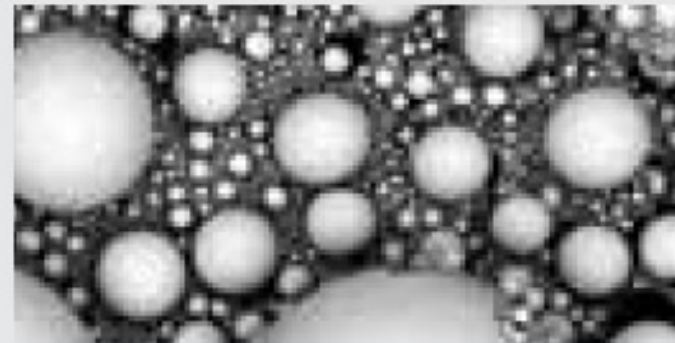




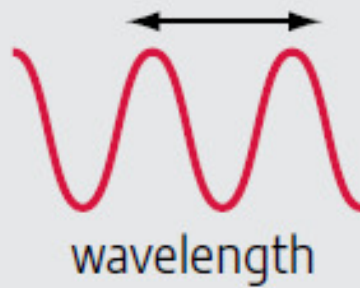
high frequency



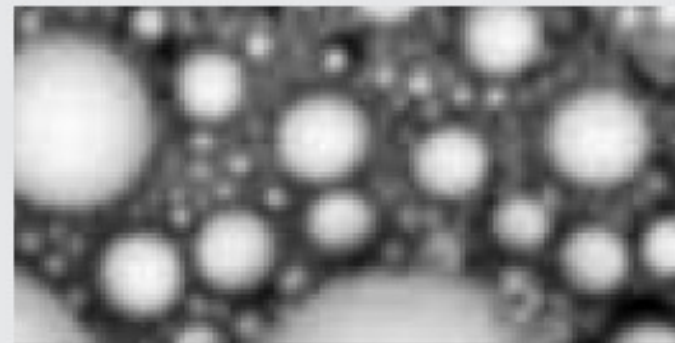
good resolution



low frequency



poor resolution

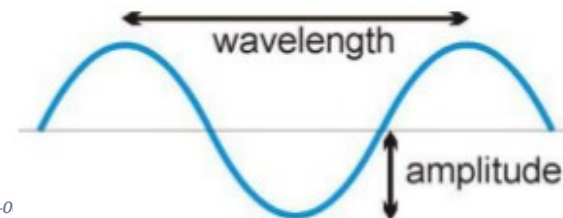




Electron Microscopy

- An electron microscope is a microscope that uses **accelerated electrons as a source of illumination.**
- Electron microscopes were developed due to the limitations of Light Microscopes which are limited by the physics of light.
- In the early 1930's there was a scientific desire to see the fine details of the interior structures of organic cells (nucleus, mitochondria...etc.).
- This required **>10000x** magnifications which could not be achieved by simple light/optical microscopy.
- Because the **wavelength of an electron can be up to 100,000 times shorter** than that of visible light photons, the electron microscope has a **higher resolving power** than a light microscope and can reveal the structure of smaller objects.

$\lambda = \sqrt{150/V}$, where
 λ - wavelength in Angstroms,
V- accelerating voltage in volts



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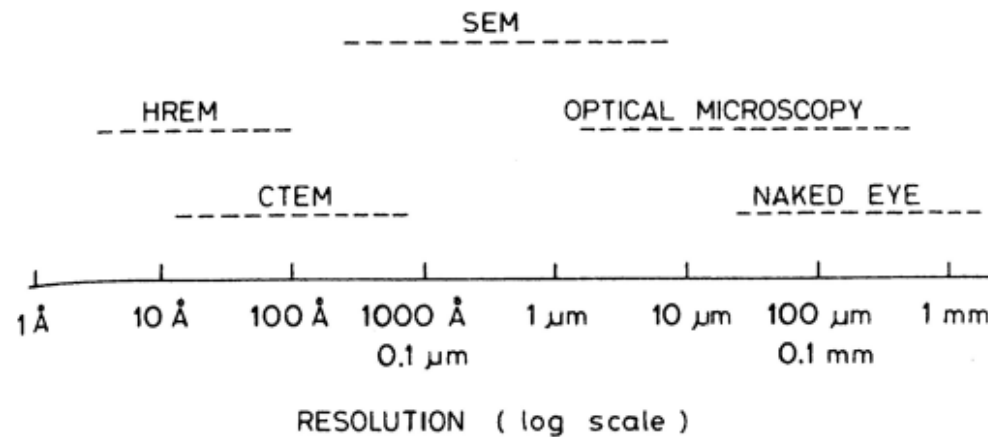
Fundamentals of Electron Microscopy

Scanning electron
microscopy (SEM):

For studying the texture, topography and surface
feature, resolution ~ 10 nm

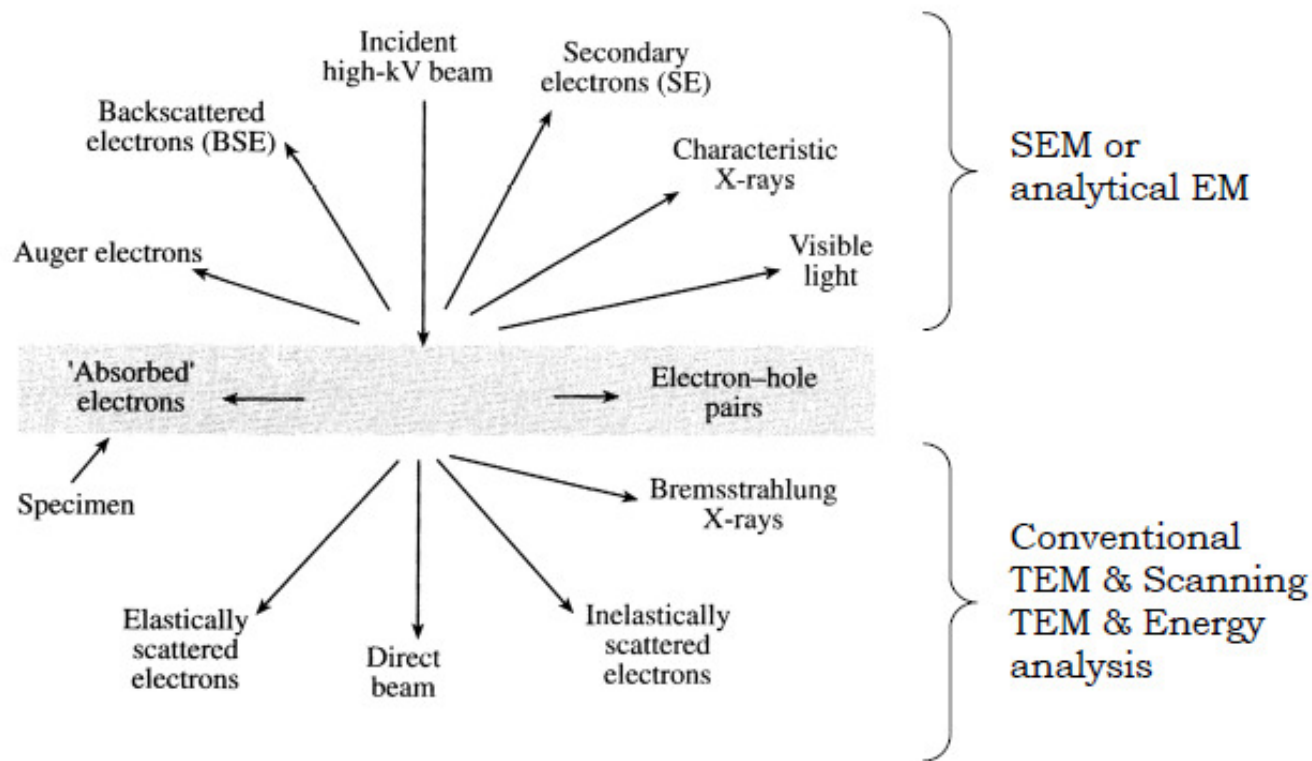
Transmission electron
microscopy (TEM):

Lattice imaging, resolution < 0.2 nm





Interaction of Electron with Samples



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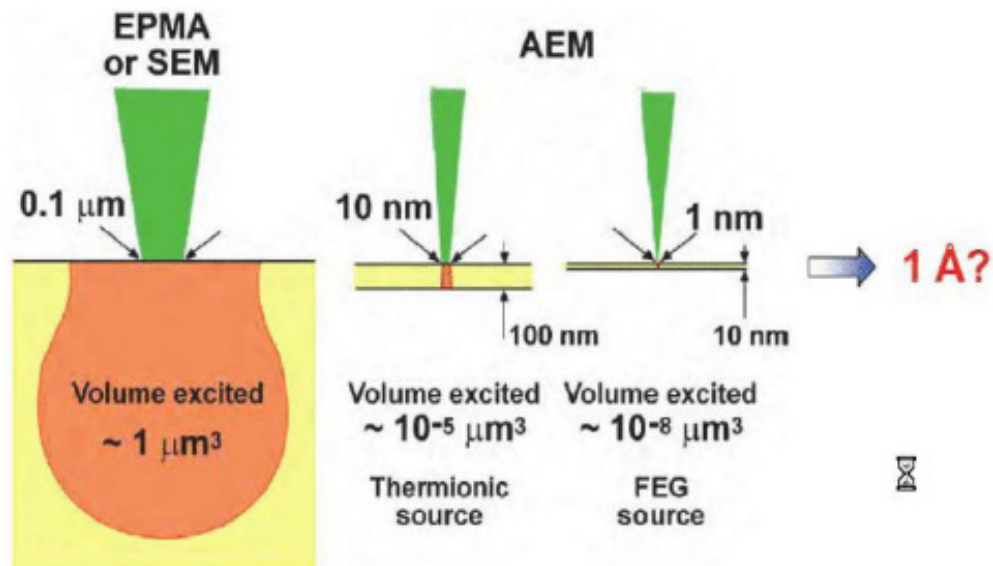
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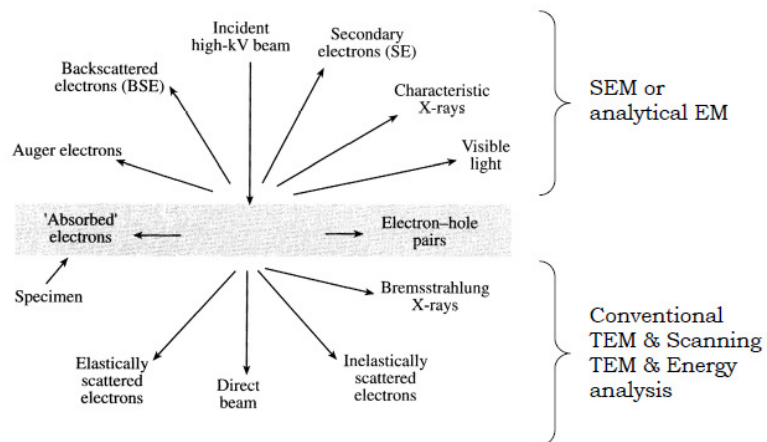


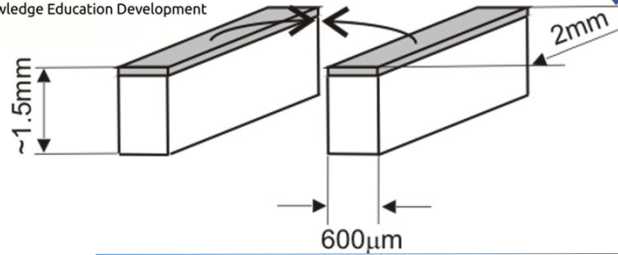
Comparing SEM and TEM

	TEM	SEM
<i>Electron Beam</i> ▶	Broad, static beams	Beam focused to fine point; sample is scanned line by line
<i>Voltages Needed</i> ▶	TEM voltage ranges from 60-300,000 volts	Accelerating voltage much lower; not necessary to penetrate the specimen
<i>Interaction of the beam electrons</i> ▶	Specimen must be very thin	Wide range of specimens allowed; simplifies sample preparation
<i>Imaging</i> ▶	Electrons must pass through and be transmitted by the specimen	Information needed is collected near the surface of the specimen
<i>Image Rendering</i> ▶	Transmitted electrons are collectively focused by the objective lens and magnified to create a real image	Beam is scanned along the surface of the sample to build up the image



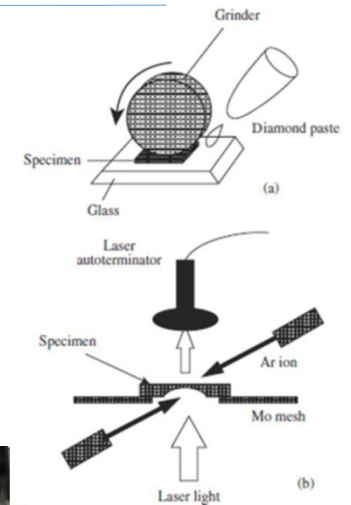
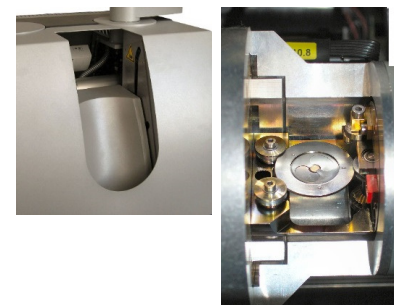
Spatial resolution \uparrow \rightarrow Analytical sensitivity \downarrow





Parameters

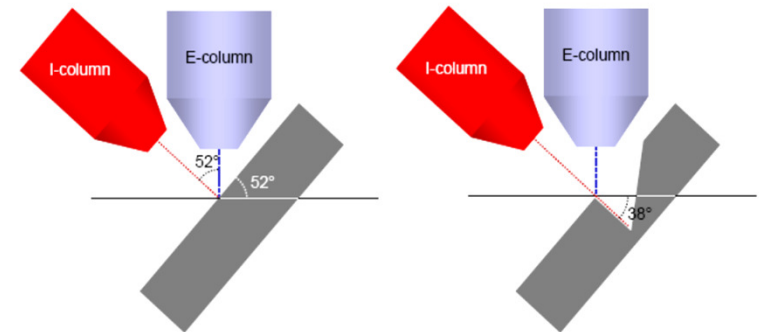
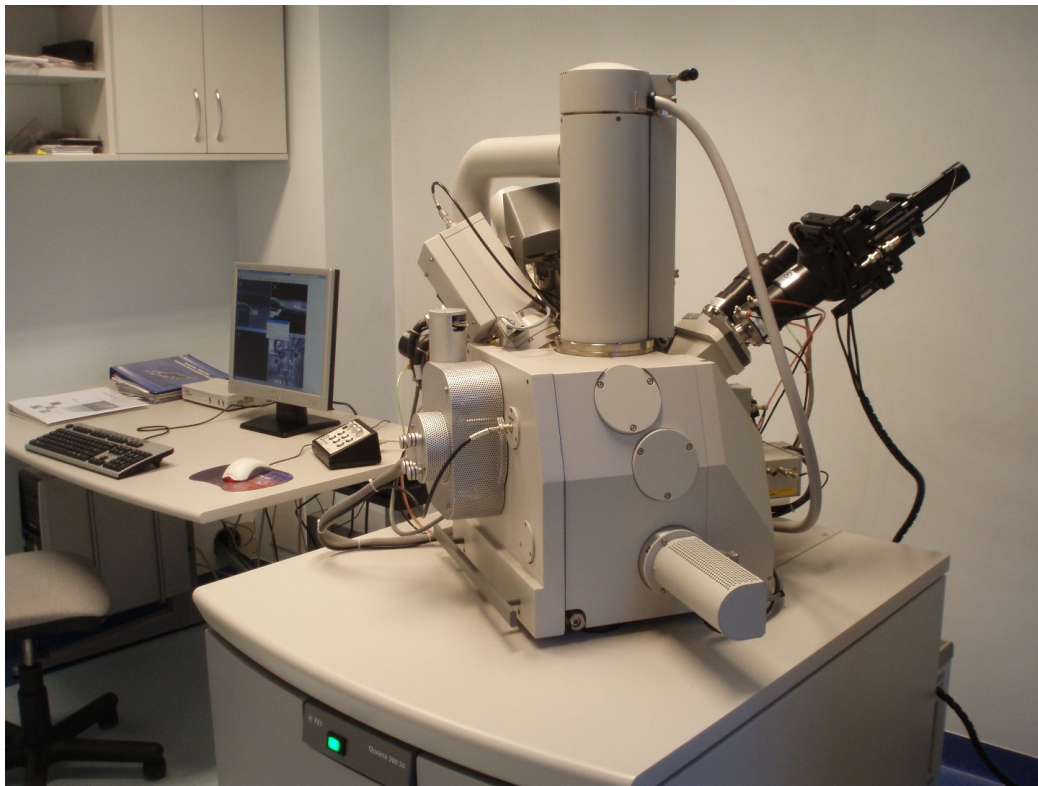
- Bench top system
- Two opposite arranged ion guns
- Ion energy: 1 keV to 10 keV
- Gun tilt: $\pm 45^\circ$ (each gun)
- Sample tilt: 0° to 180°
- Milling angle: 0° to 90°



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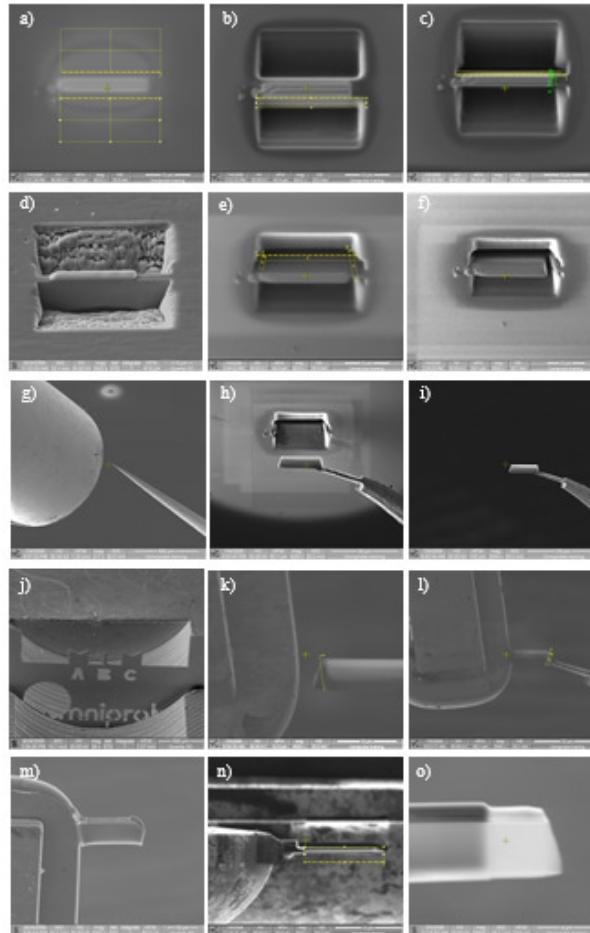
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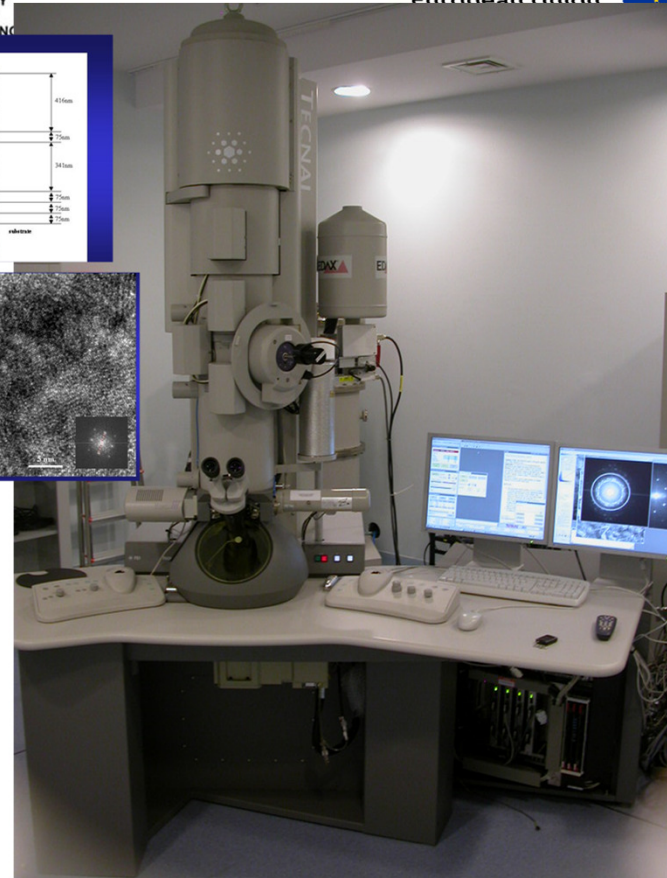
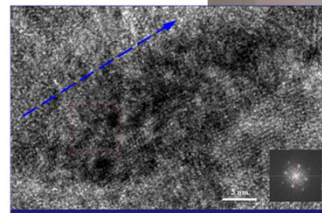
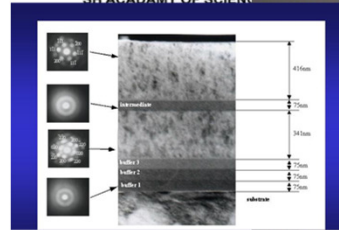
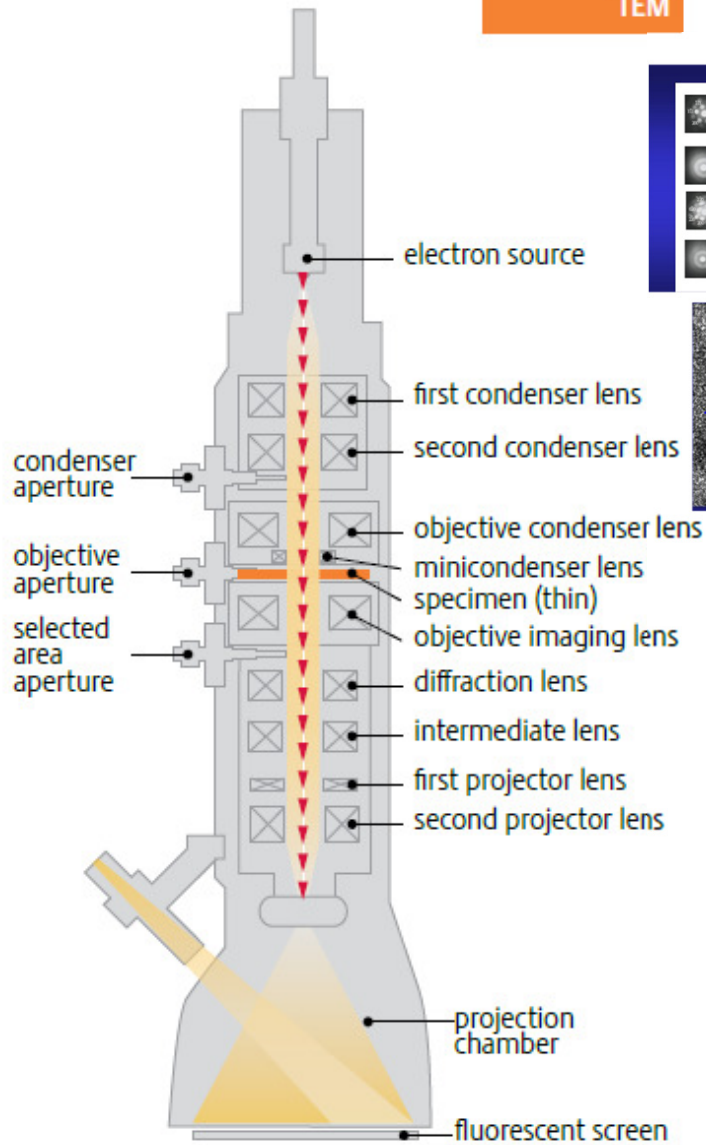
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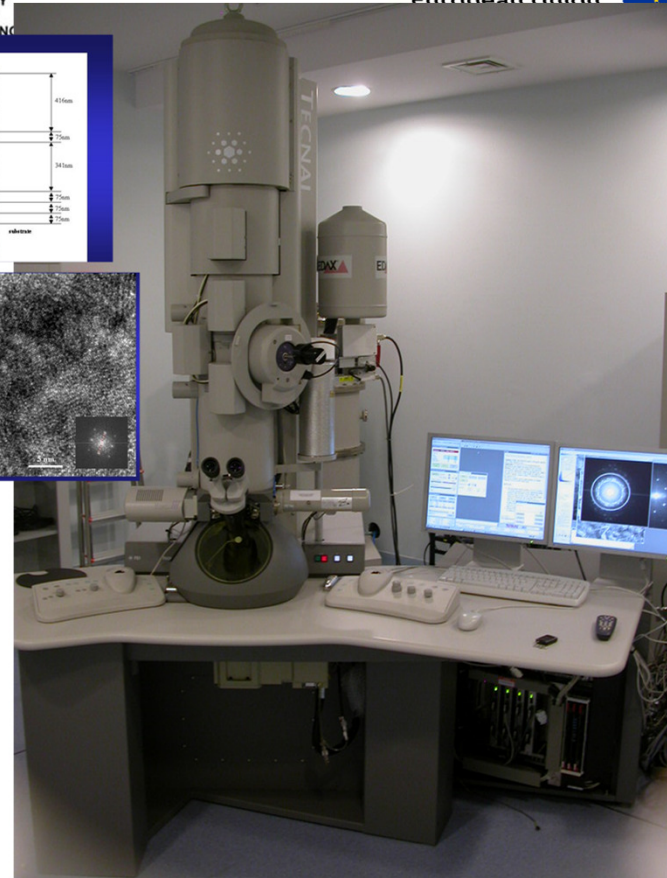
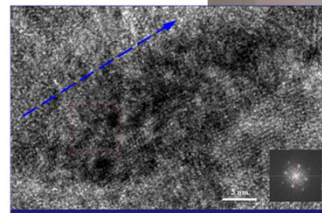
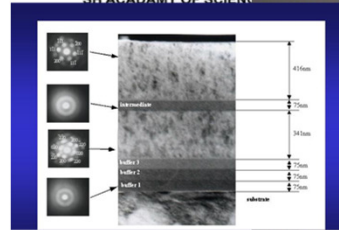
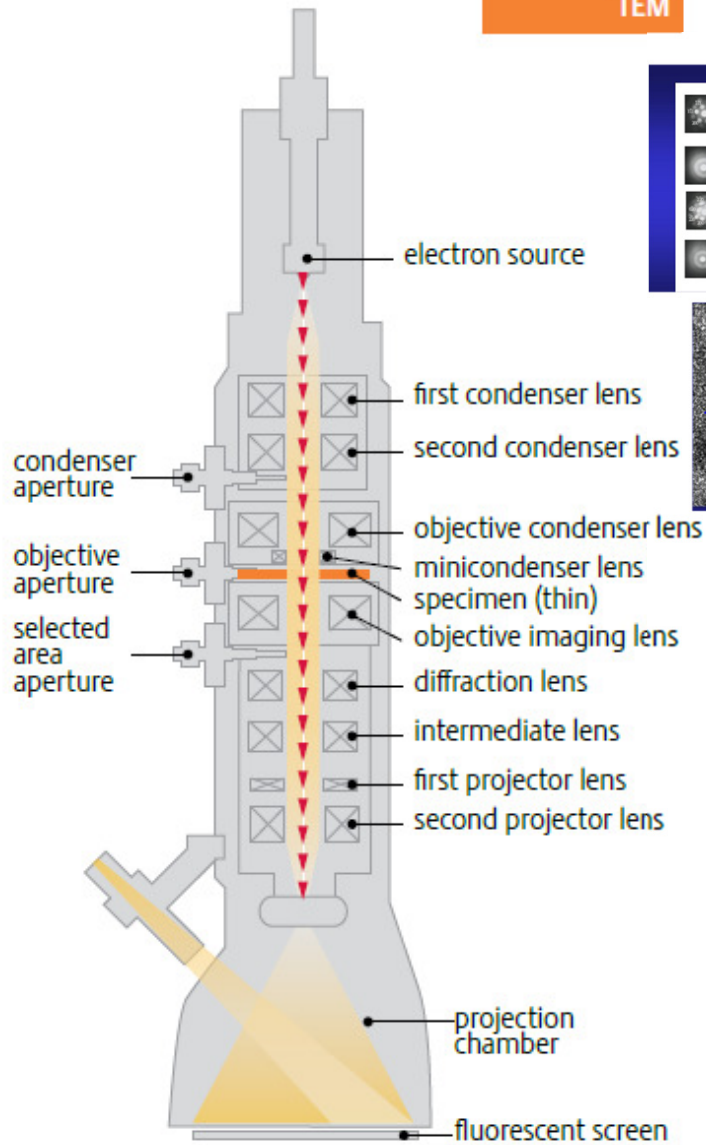


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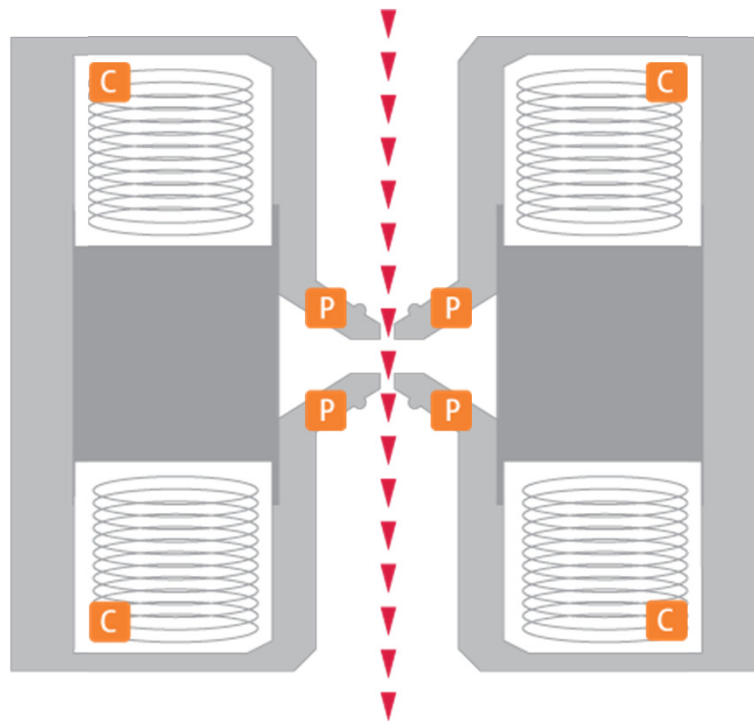
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CORE TECHNOLOGY: Electromagnetic Lenses

electron beam



C electrical coil

P soft iron pole piece

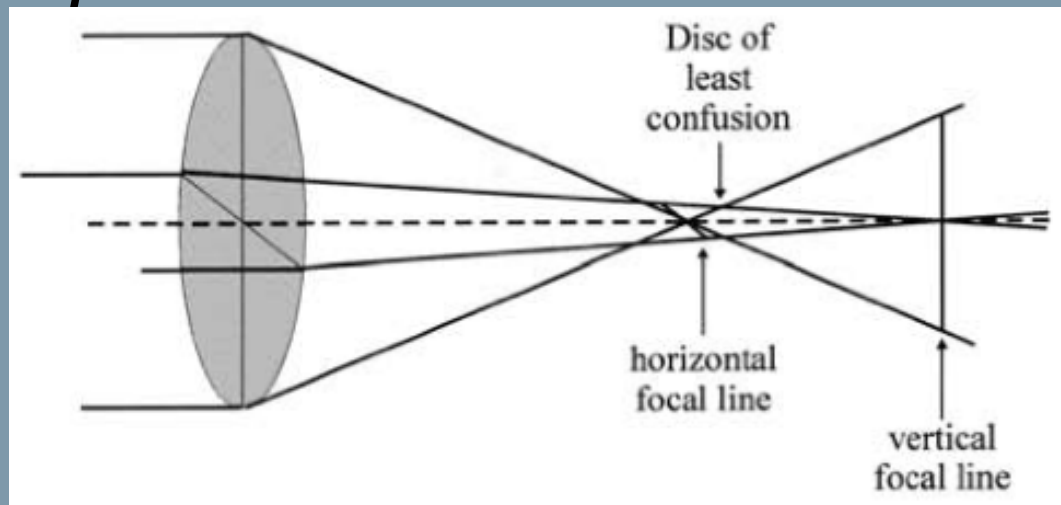
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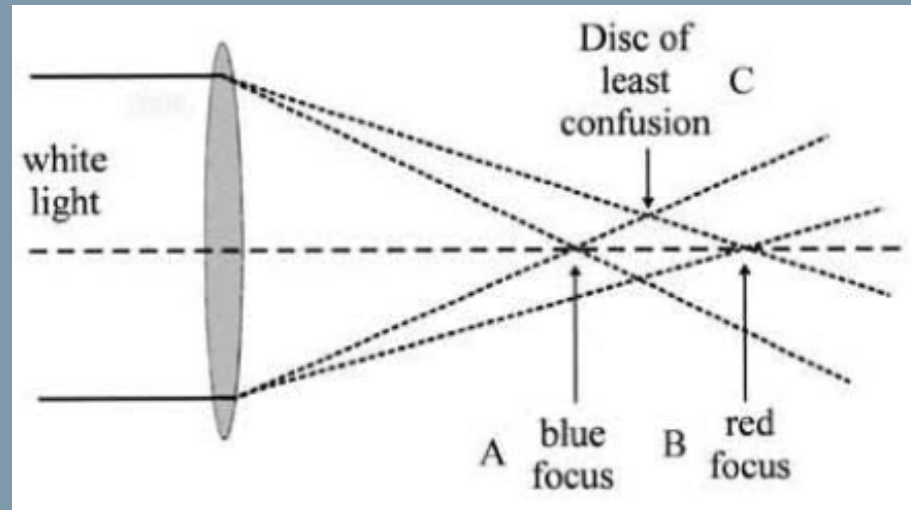
spherical aberration



- arises because a simple lens is more powerful at the edge than at the centre
- is not a problem with glass lenses (can be ground to shape)
- disc of *minimum confusion* results instead of point focus:
- is *not correctable* for electromagnetic lenses



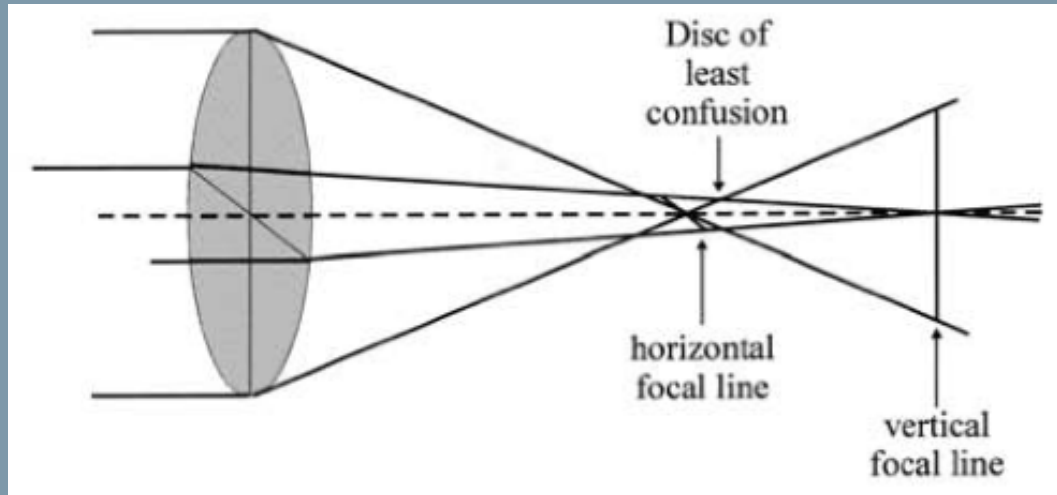
chromatic aberration



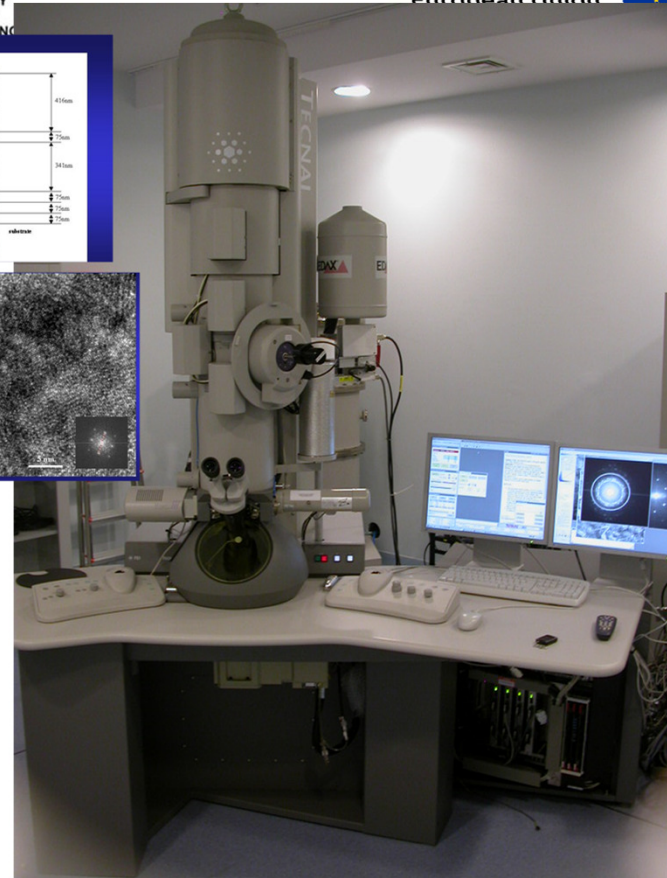
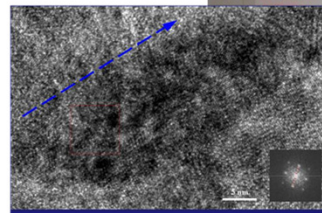
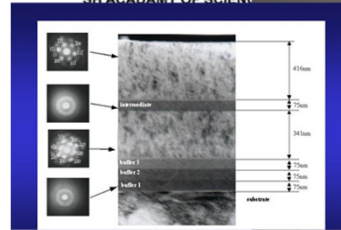
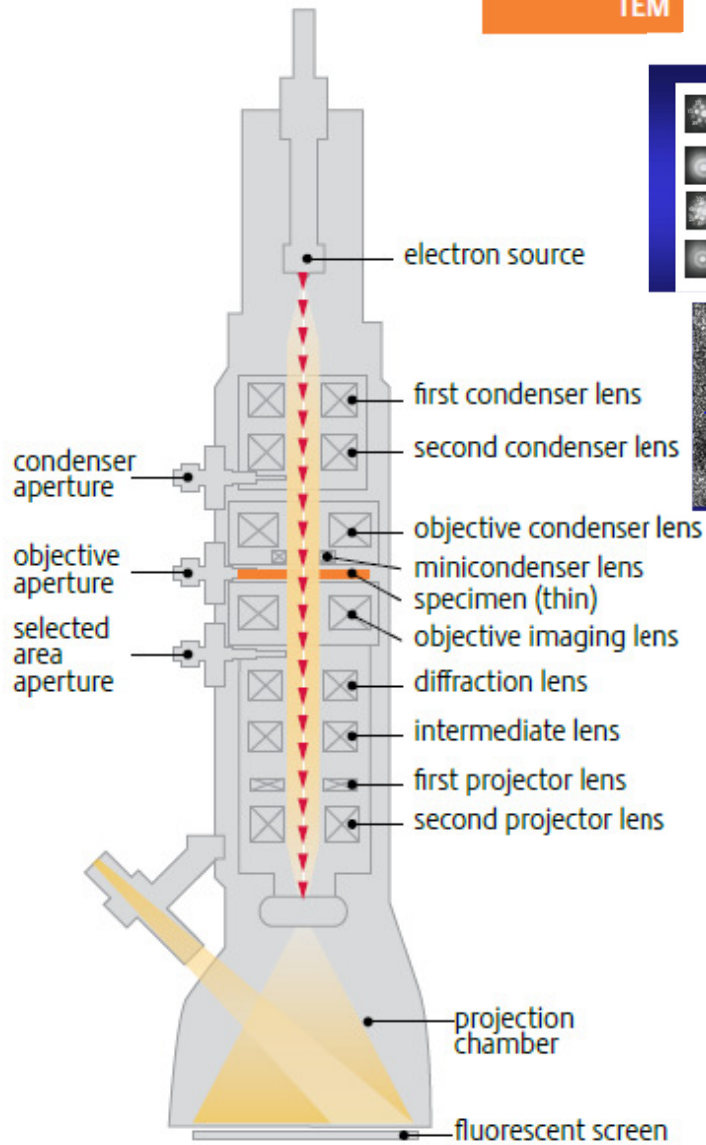
- light of different λ brought to different focal positions
- λ for electrons can be controlled by fixed KV and lens currents
- but λ of electrons can *change* by interaction with specimen !
- rule of thumb: **resolution \geq (specimen thickness)/10**



astigmatism

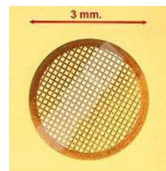
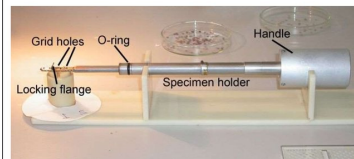
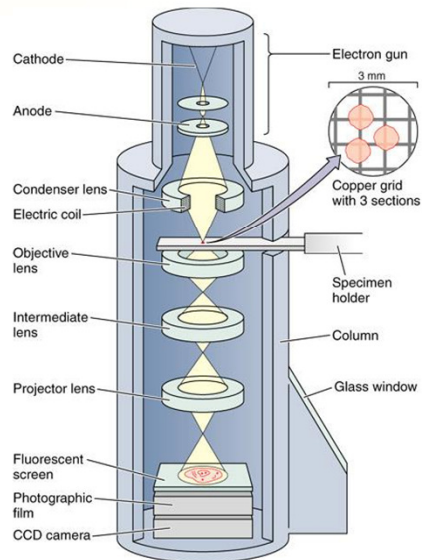


- arises when the lens is more powerful in one plane than in the plane normal to it
- causes points to be imaged as short lines, which 'flip' through 90 degrees on passing through 'focus' (minimal confusion)

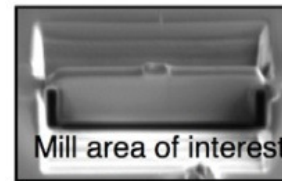


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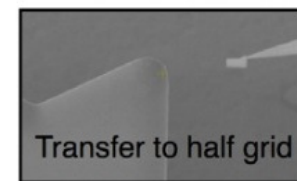
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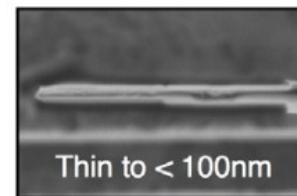
Supporting grid for TEM specimens



Mill area of interest



Transfer to half grid

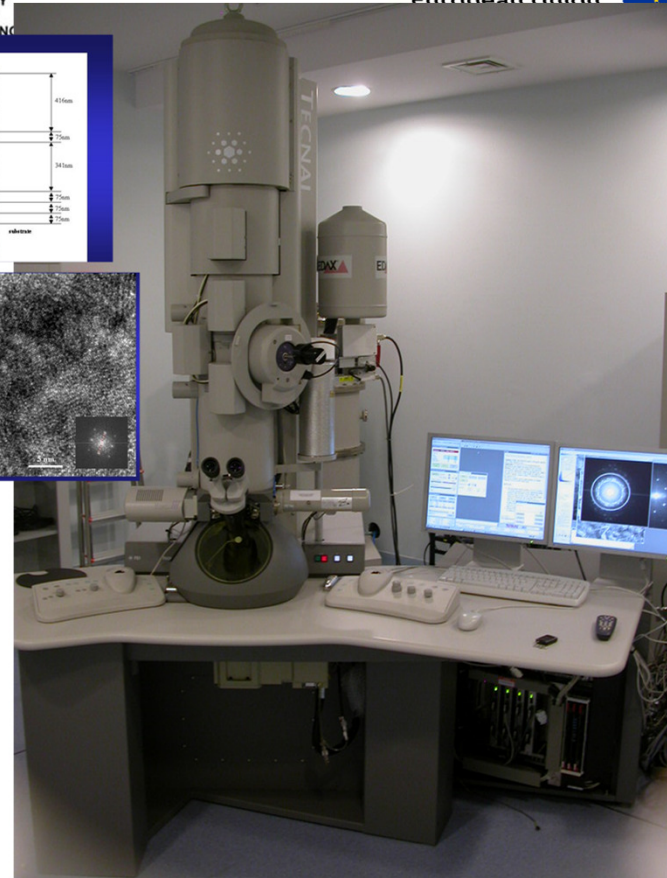
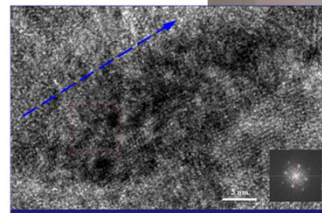
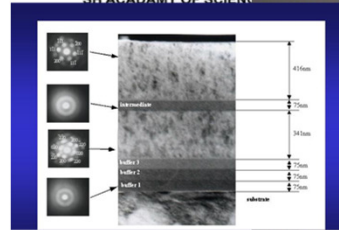
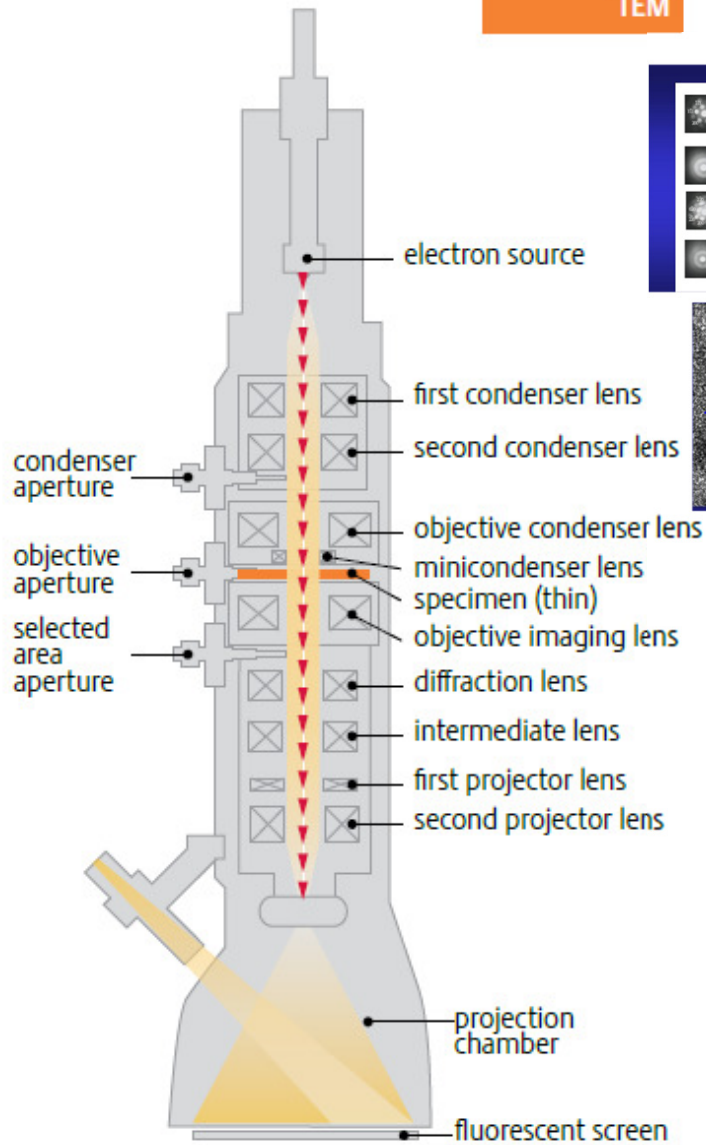


Thin to < 100nm

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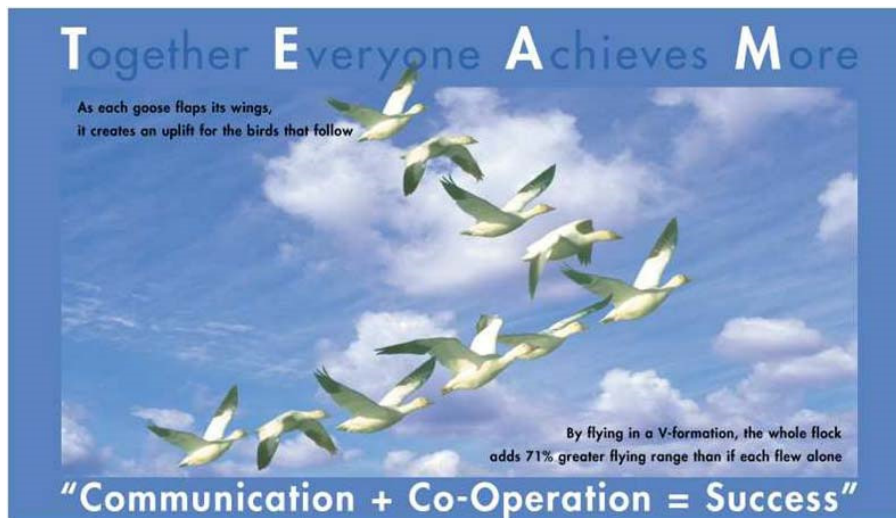
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Dr hab. eng. Jürgen Lackner

Coatings deposition

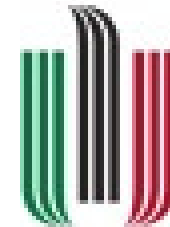
Dr hab. eng. Łukasz Major

TEM microstructure characterization

Dr hab. eng. Zbigniew Nawrat



Cardiosurgical robots



AGH

Dr hab. eng. Marcin Kot

Micromechanical analysis

Dr eng. Marcin Dyner



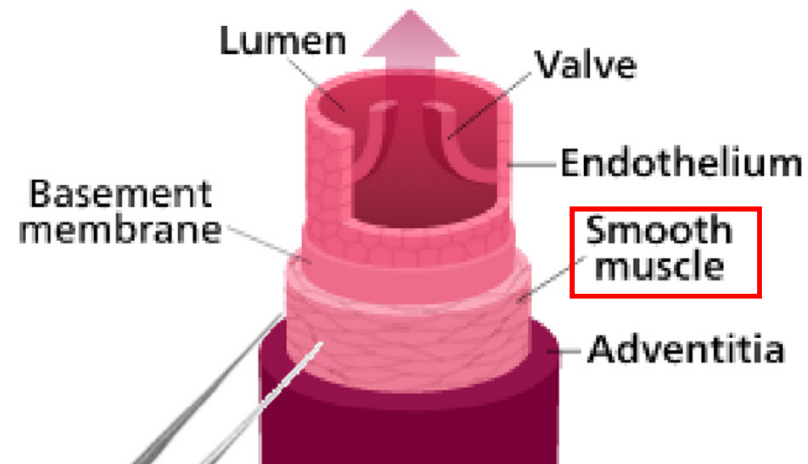
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To heart



Approach for coatings design

- Advanced deposition techniques
- Detailed microstructure characterization
- Micro- mechanical tests + finite elements modeling
- Bio- compatibility tests

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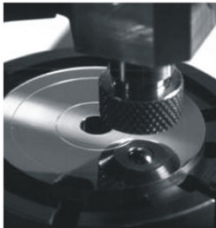


Coatings were selected by:

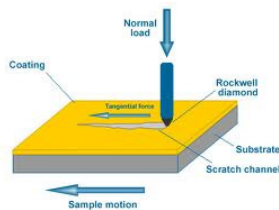
Coatings were characterized by:

Mechanical test

- Ball-on-disc wear test

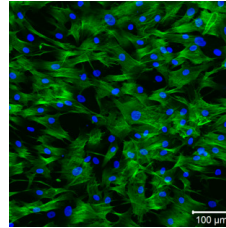


- Scratch adhesion test



Bio-compatibility test

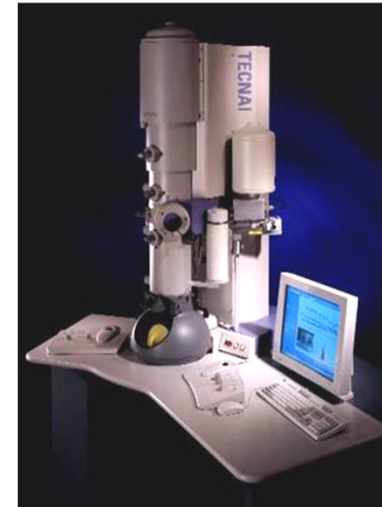
- Test by smooth muscle cells



- Confocal microscopy



Transmission Electron Microscopy



Bio-tribological tests

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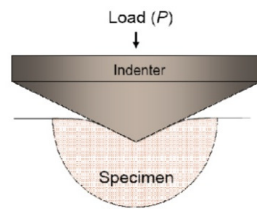
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Microstructure characterization

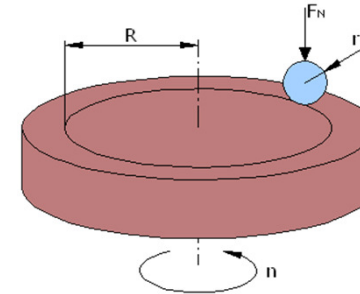
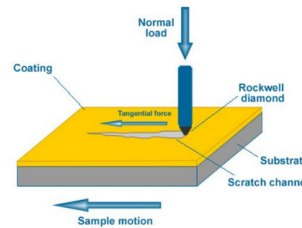


Standard, ex- situ tests

Indentation test (Hardness test)



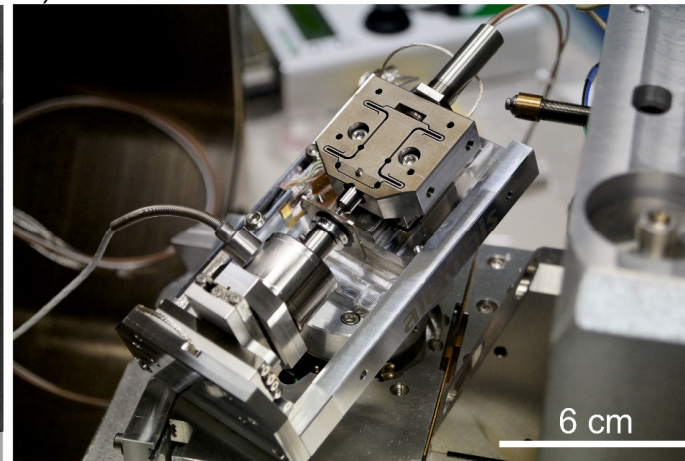
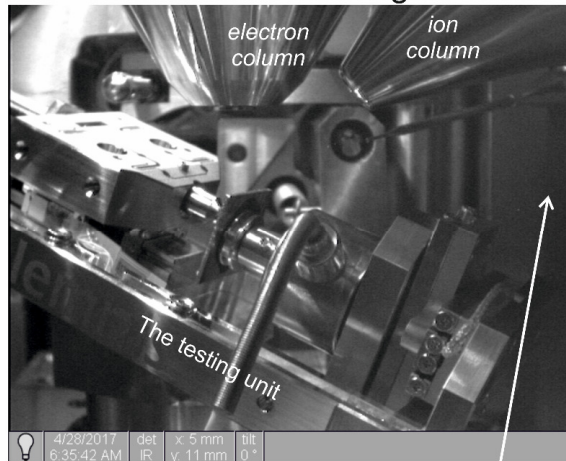
Scratch test set-up



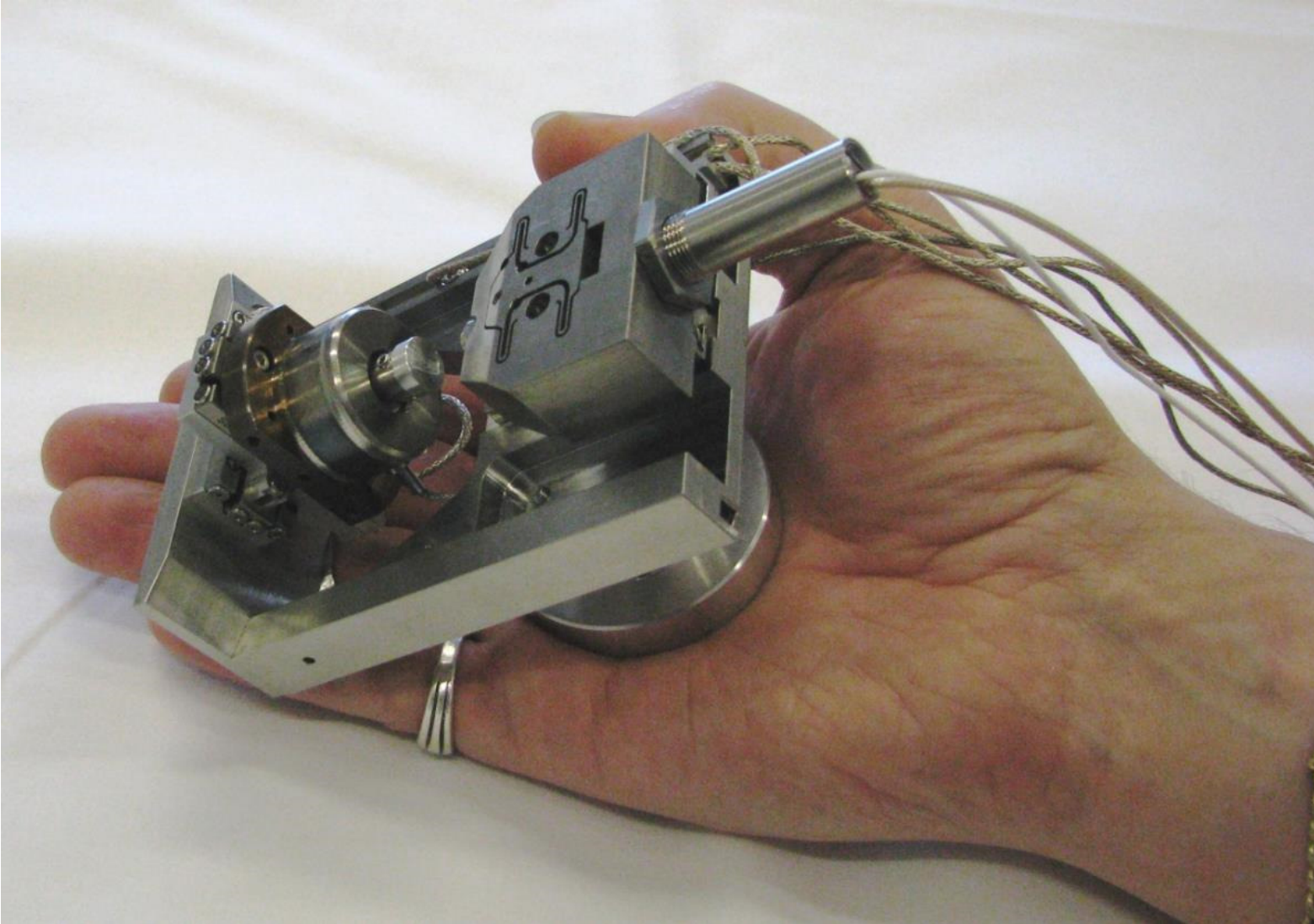
- Hardness – resistance to penetration of a hard indenter

In- situ tests

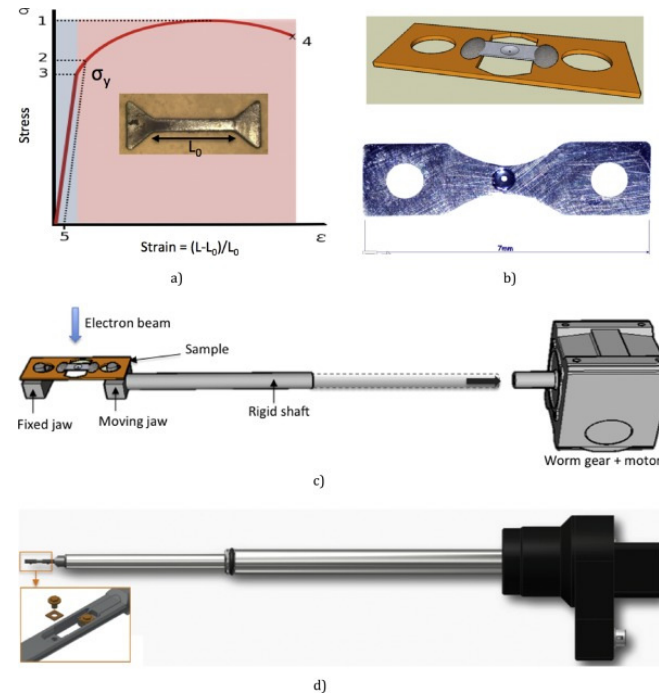
a) SEM chamber with the testing unit inside b)



vacuum chamber of the SEM



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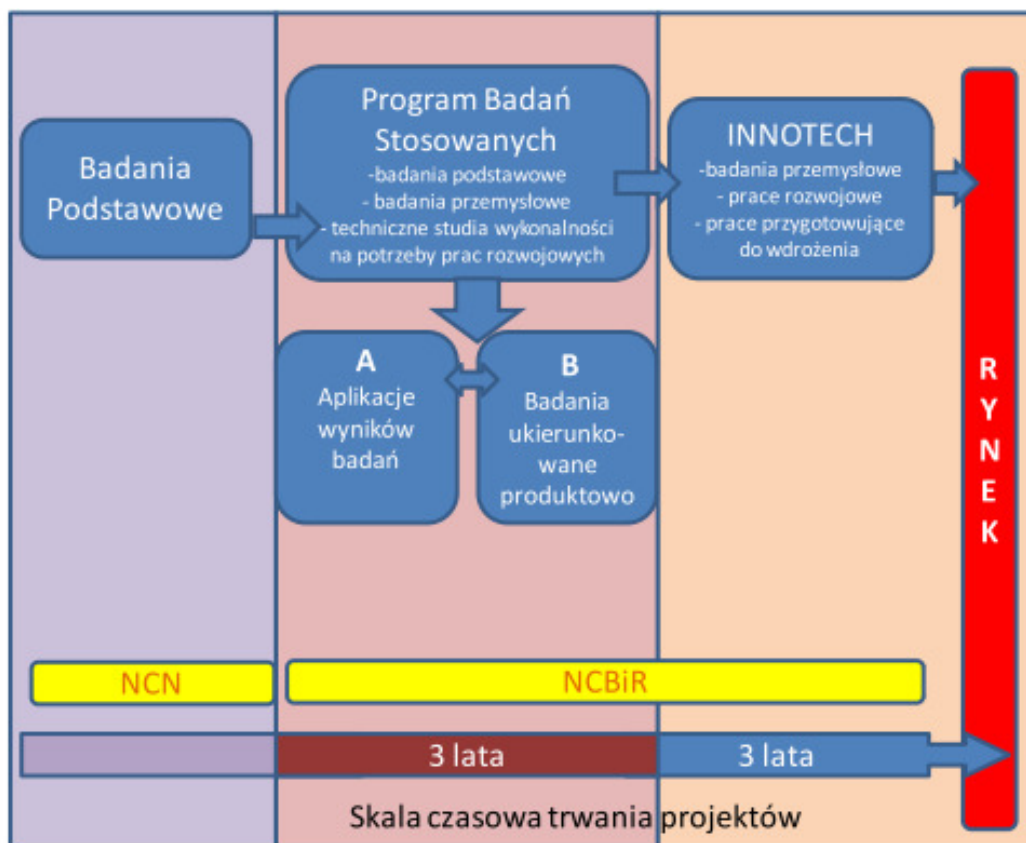
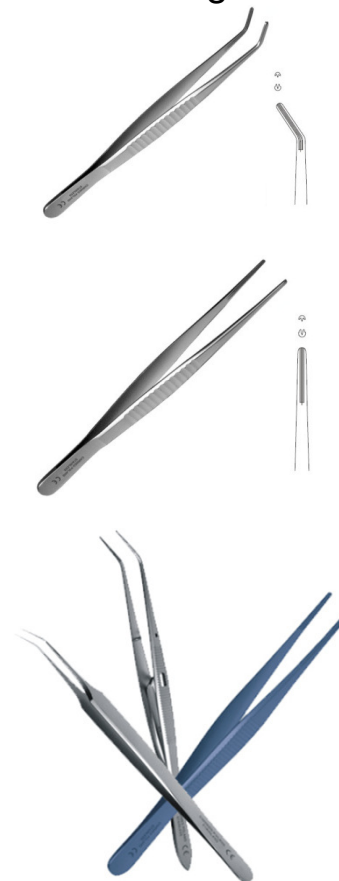
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General goal



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- Project NCN nr: 3066/B/T02/2011/40- *FINISHED*

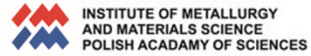
- Project NCN nr: 2012/06/M/ST8/00408- *HARMONIA- FINISHED*

- Project NCN nr: 2012/07/B/ST8/03396- *OPUS- FINISHED*

- Project NCN nr: 2014/15/B/ST8/00103- *OPUS- FINISHED*

- Project NCN nr: 2015/19/B/ST8/00942- *OPUS- in progress*

- Project NCBR, number: DZP/M-ERA.NET-2015/285/2016- *in progress*

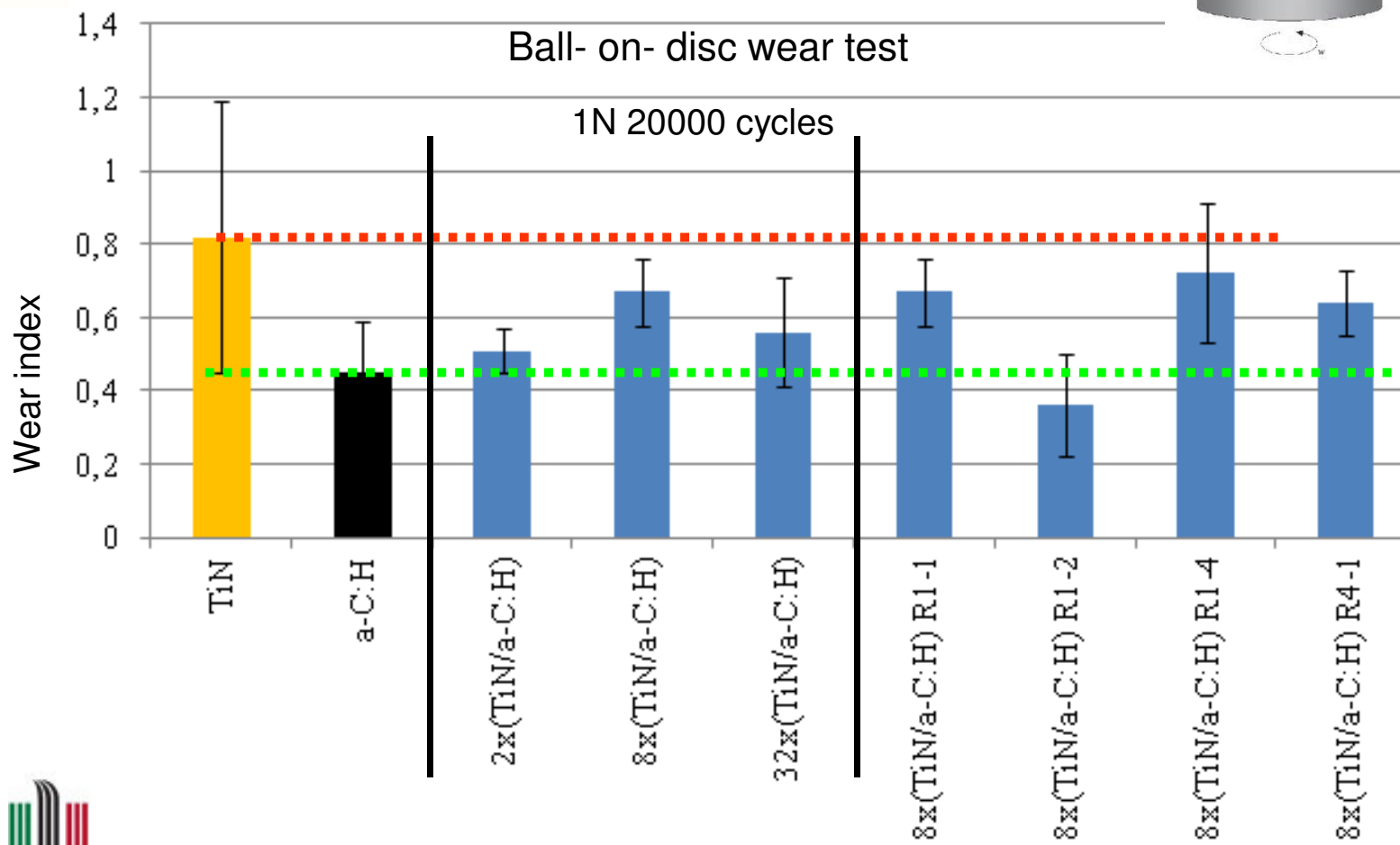
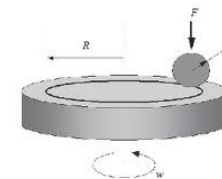


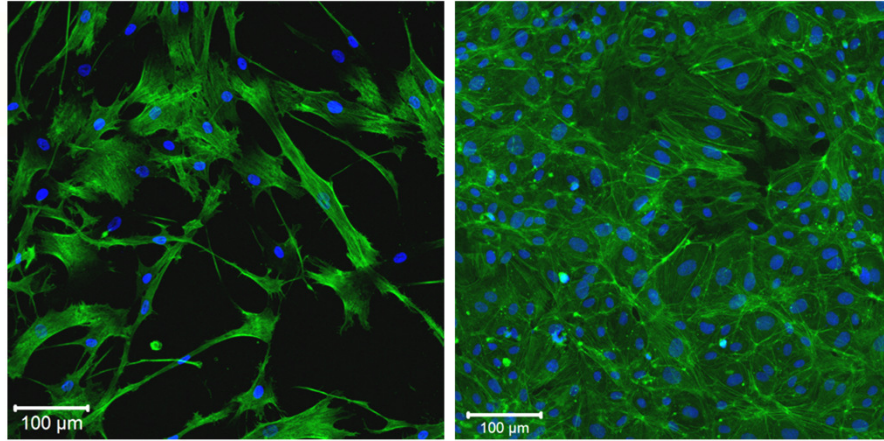
Title: Development and diagnostics of multifunctional ceramic/ hydrogenated amorphous carbon coatings for elements of pumps of ventricle assist pumps

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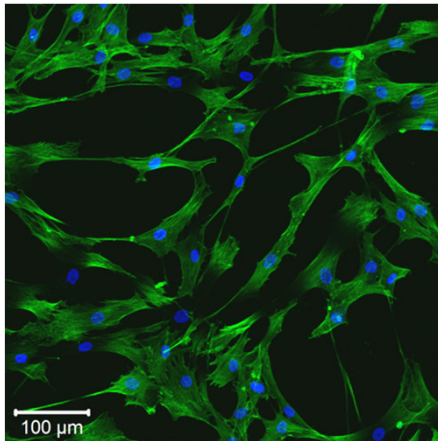
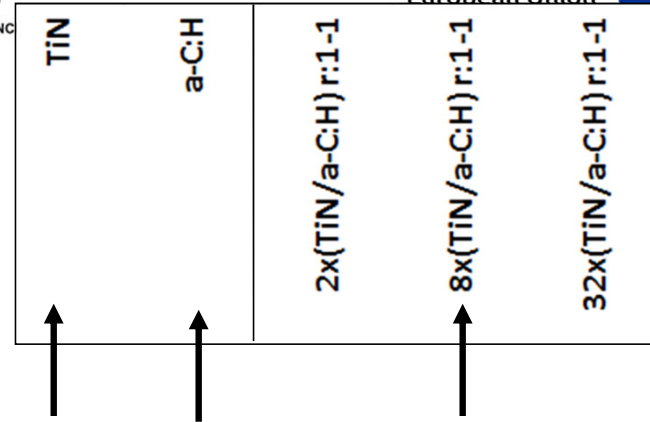
TiN

a-C:H

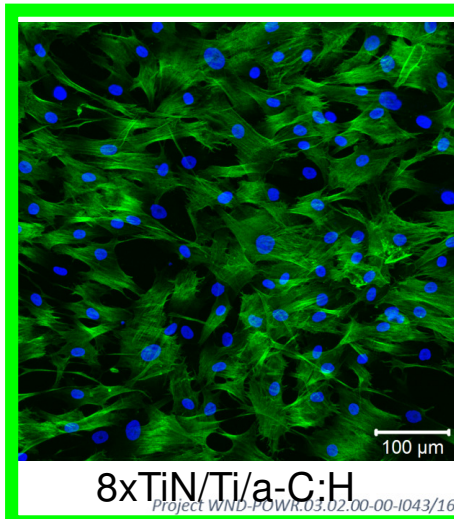
Reference coatings

Bio-compatibility test

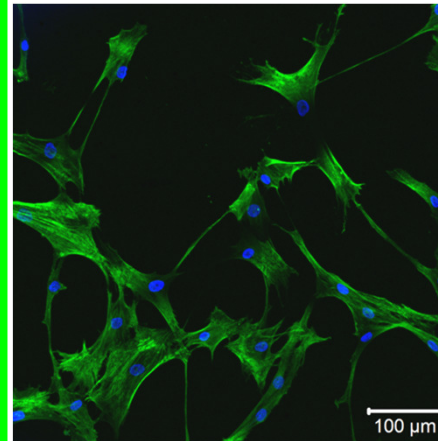
European Union



2xTiN/Ti/a-C:H



8xTiN/Ti/a-C:H



32xTiN/Ti/a-C:H

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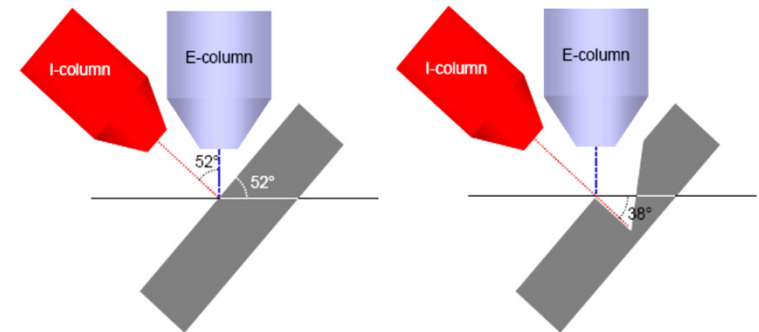
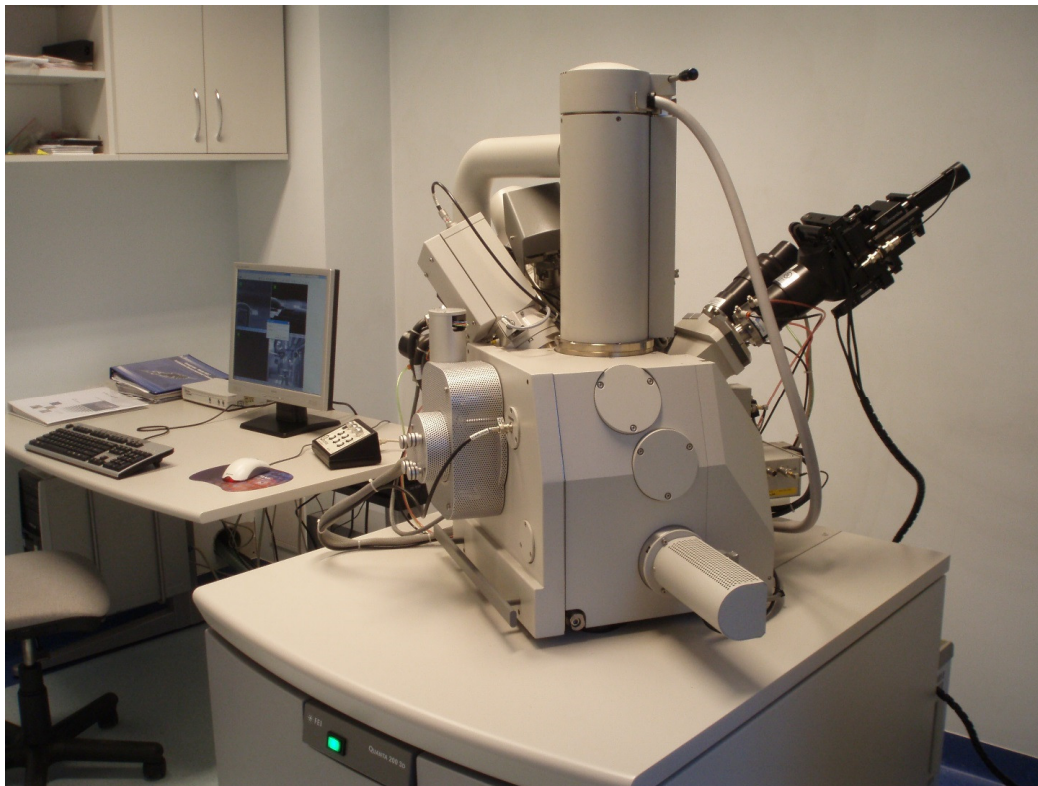
Single layer a-C:H coating

As deposited coating (Before mechanical tests)
Microstructure characterization

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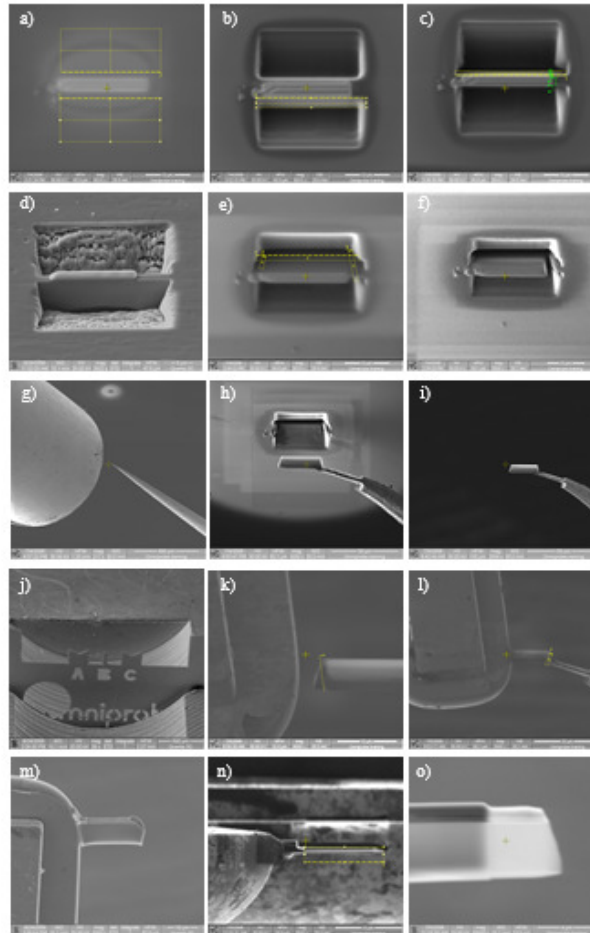
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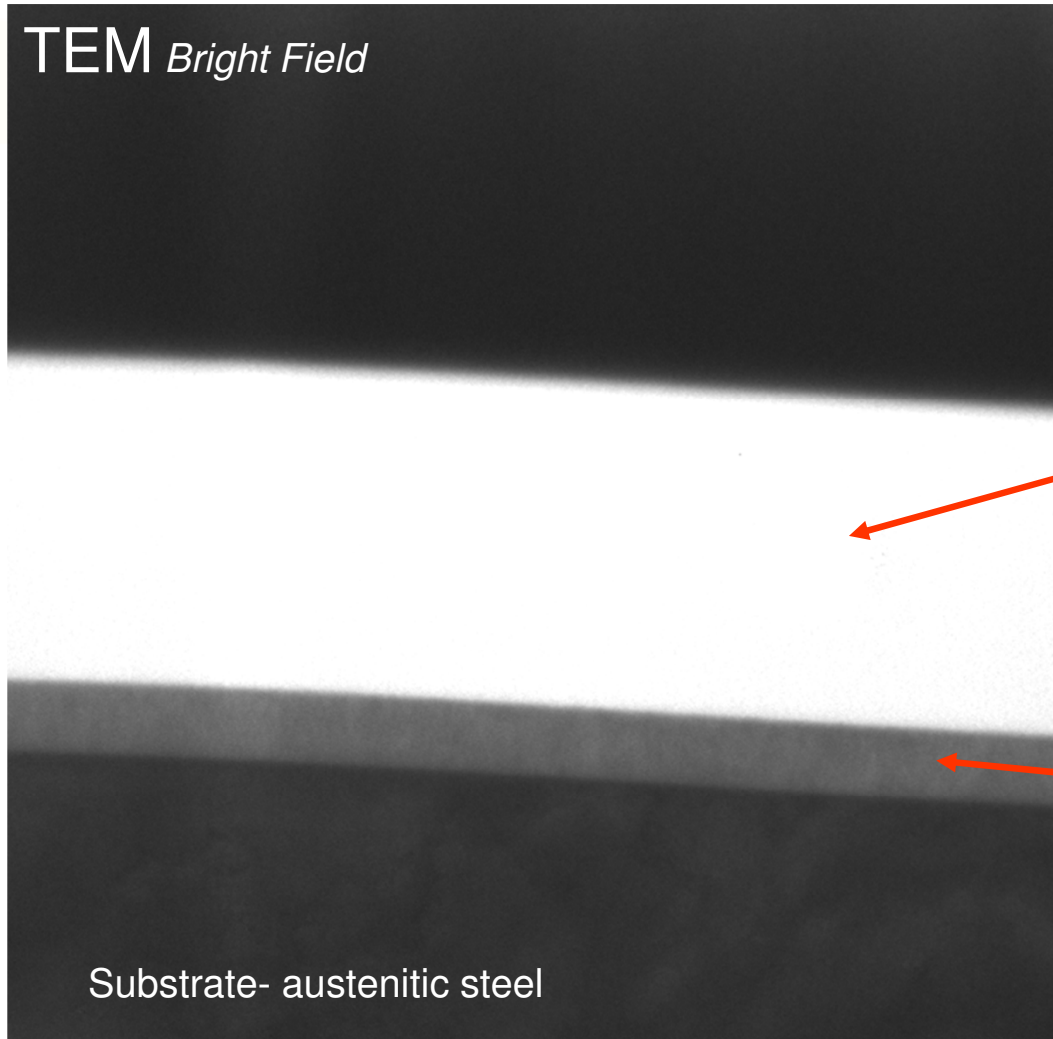
Project WND-POWR.03.02.00-00-I043/16

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TEM *Bright Field*

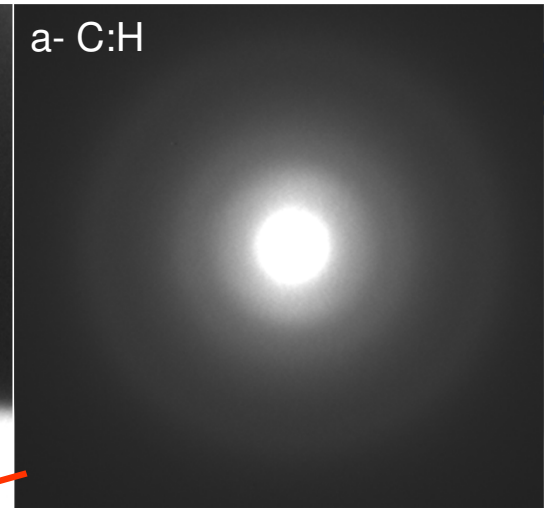


Substrate- austenitic steel

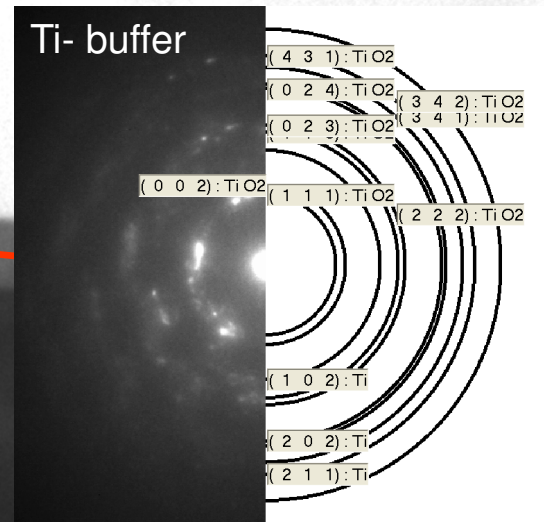


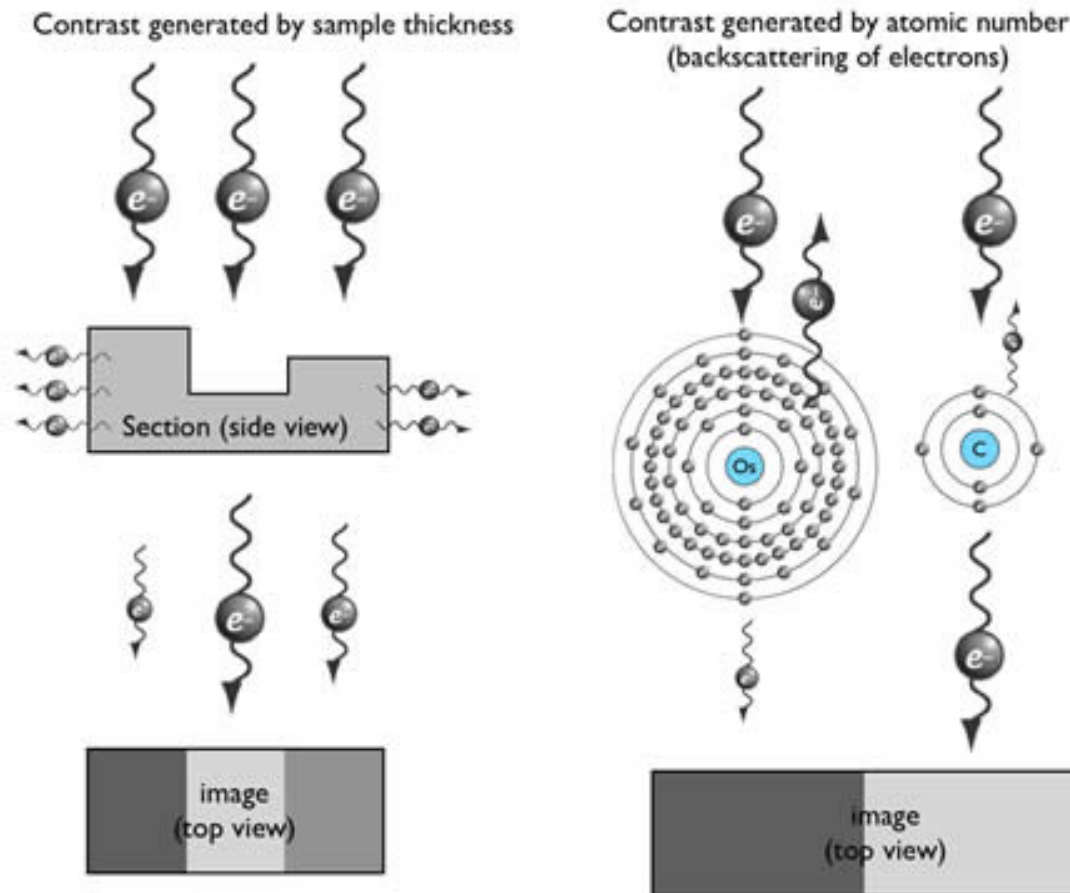
500 nm

a- C:H



Ti- buffer





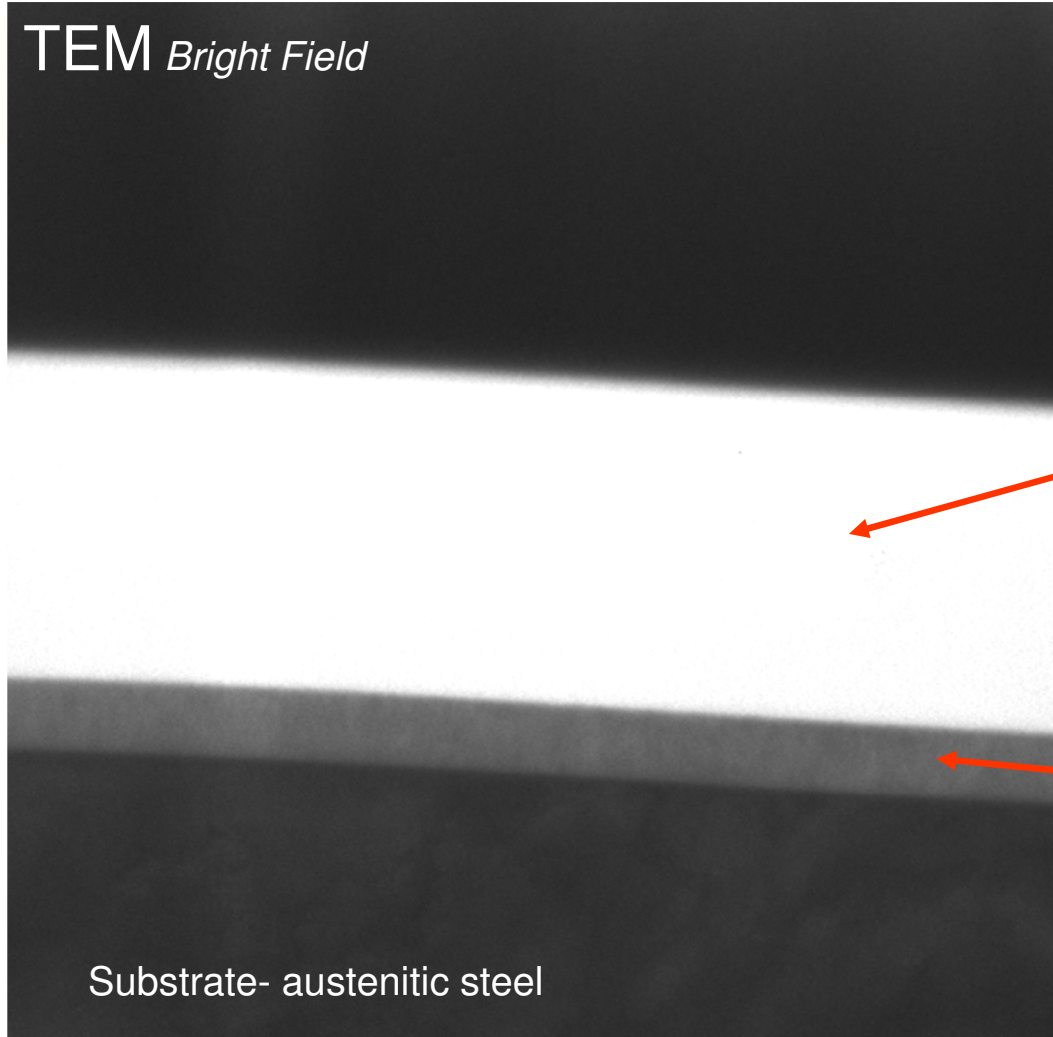
Project WND-POWR.03.02.00-00-1043/16

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TEM *Bright Field*

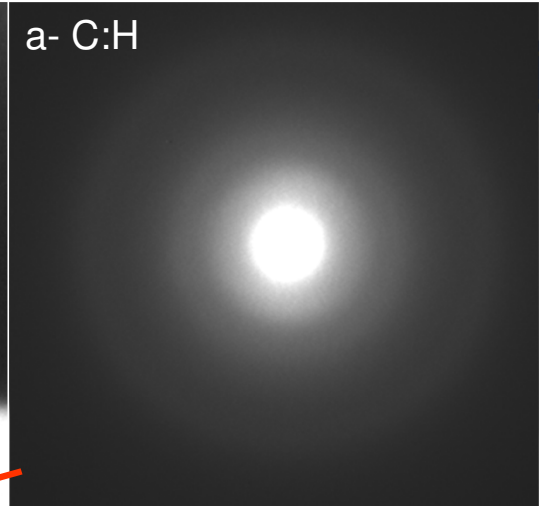


Substrate- austenitic steel

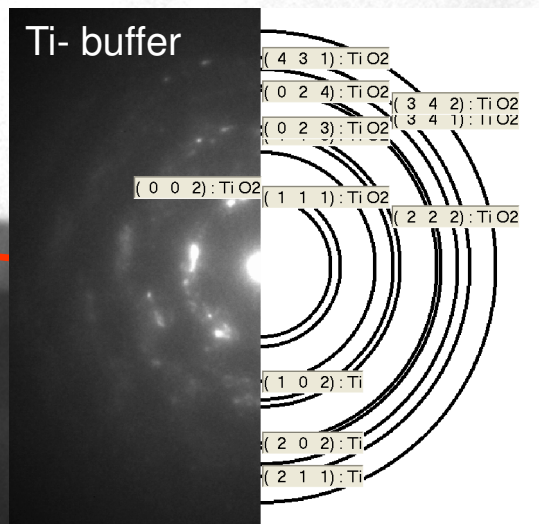


500 nm

a- C:H



Ti- buffer



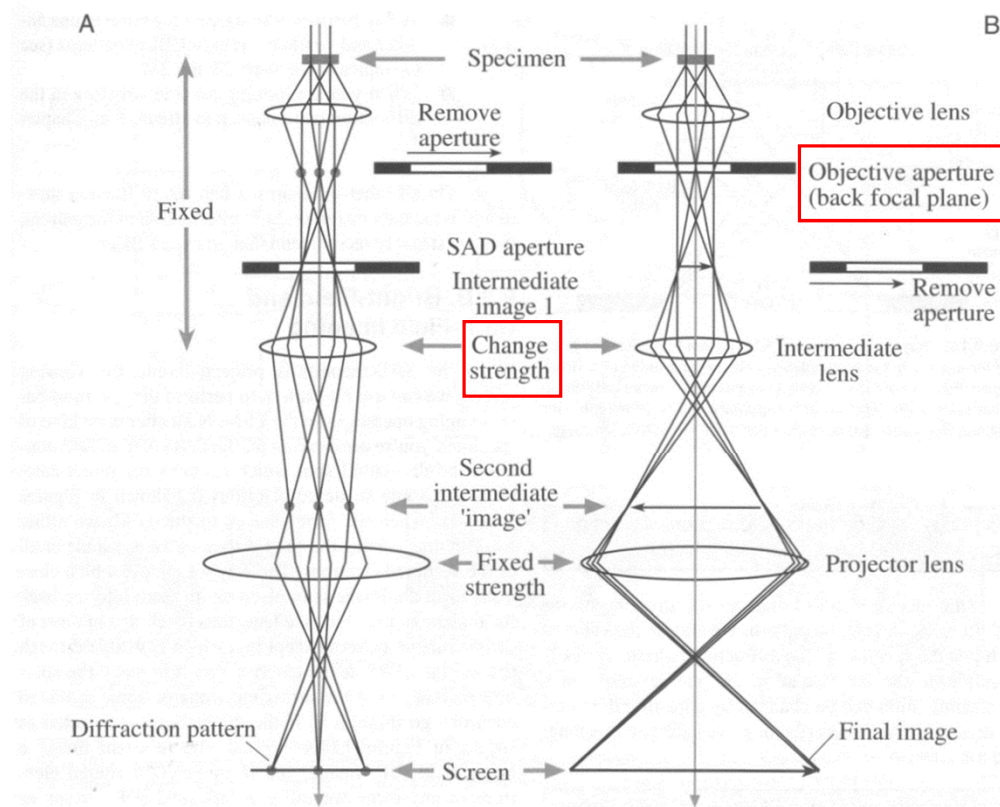
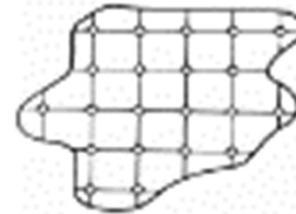


Figure 9.12. The two basic operations of the TEM imaging system involve (A) projecting the diffraction pattern on the viewing screen and (B) projecting the image onto the screen. In each case the intermediate lens selects either the back focal plane or the image plane of the objective lens as its object.

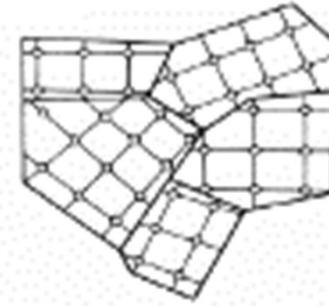
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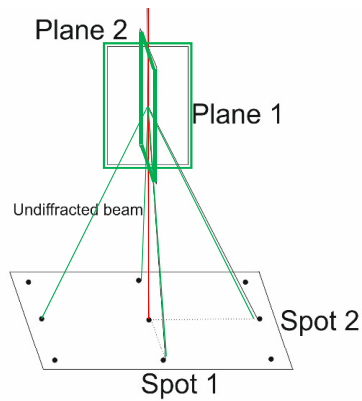
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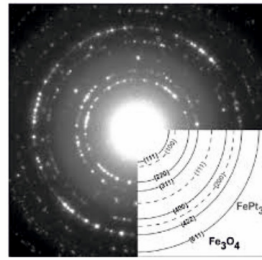
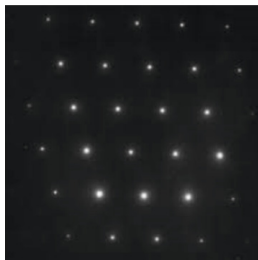
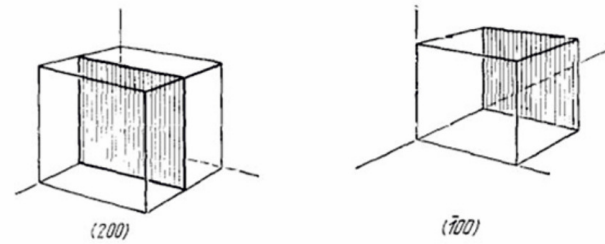
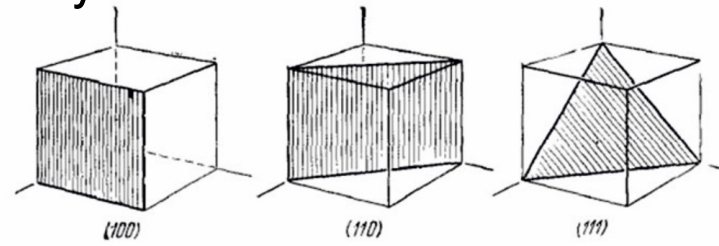
Single crystal



Poly crystal



Miller indices of major crystal planes of the regular system





Single layer a-C:H coating

Coating after mechanical tests (Ball-on-disc 1N; 2000 cycles)
Microstructure characterization

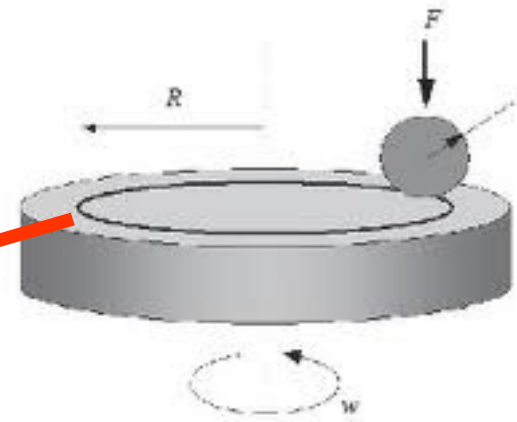
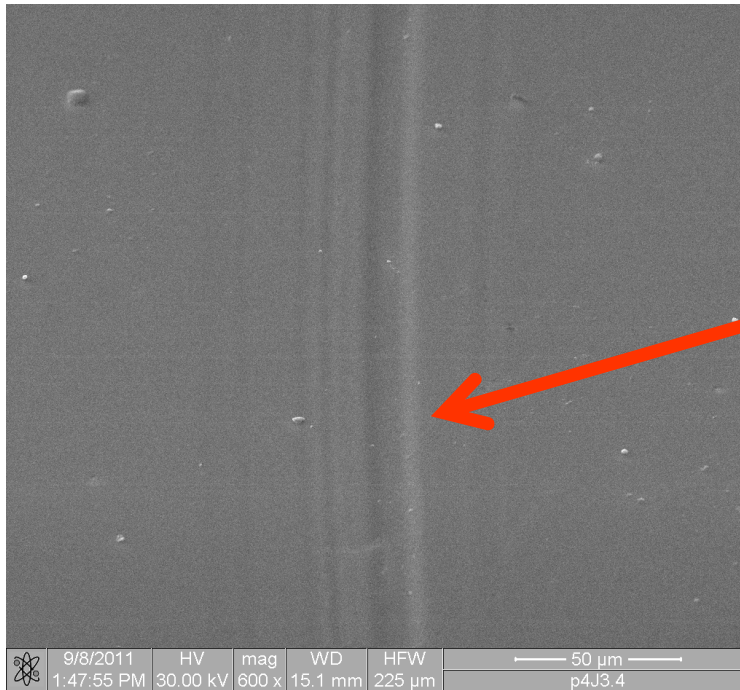
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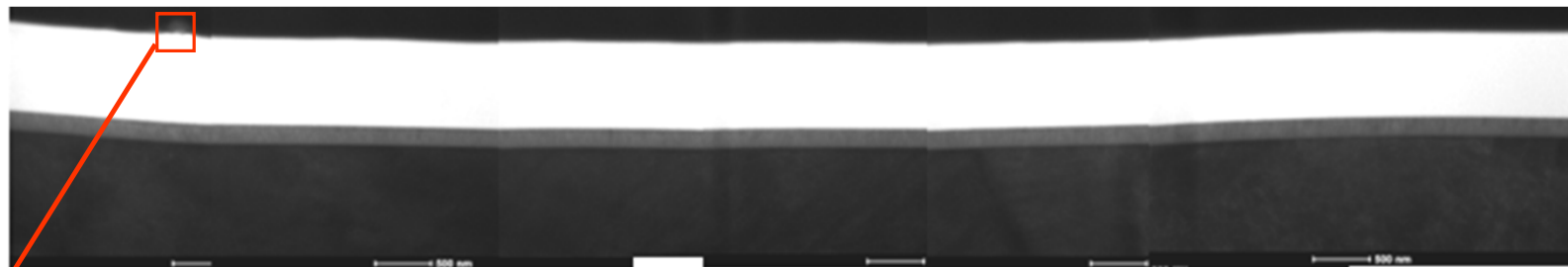
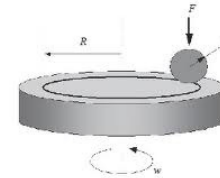


SEM

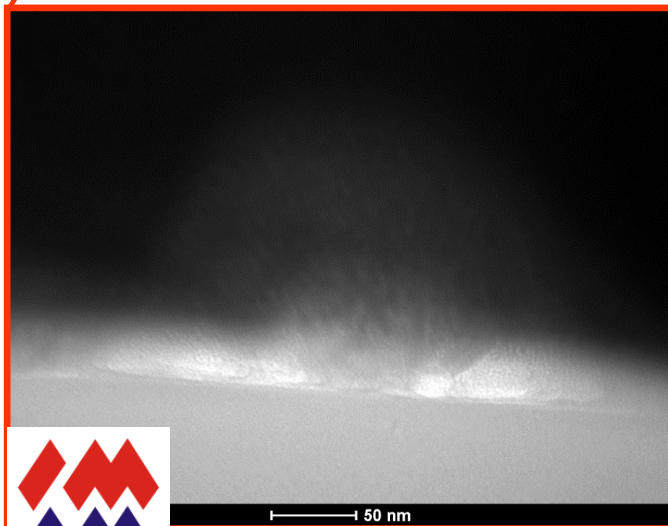




TEM *Bright Field;* *Coating after mechanical test*



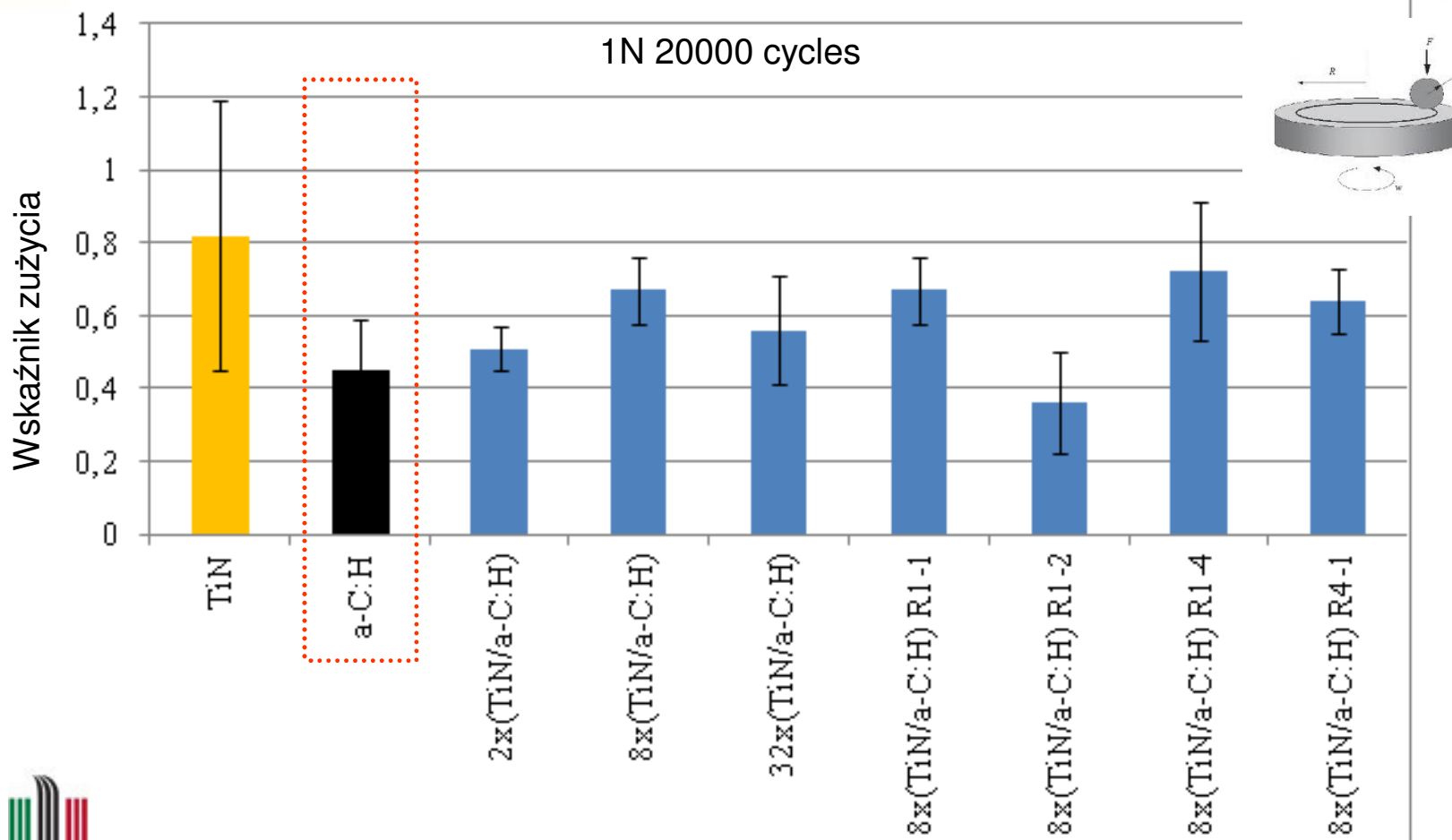
500 nm



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Single layer TiN coating

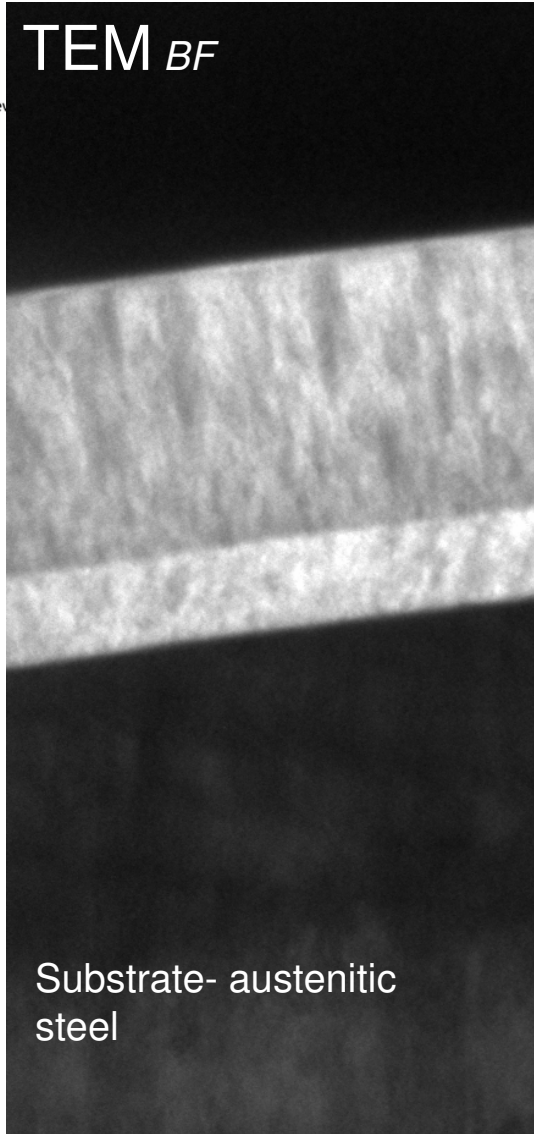
As deposited coating (Before mechnaical tests)
Microstructure characterization

Project WND-POWR.03.02.00-00-1043/16

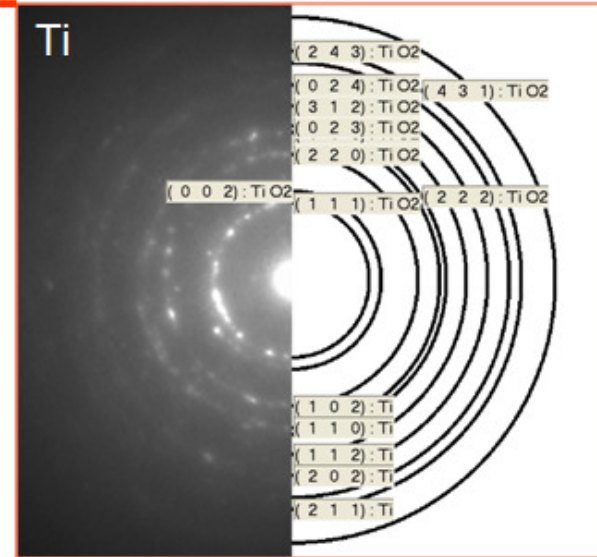
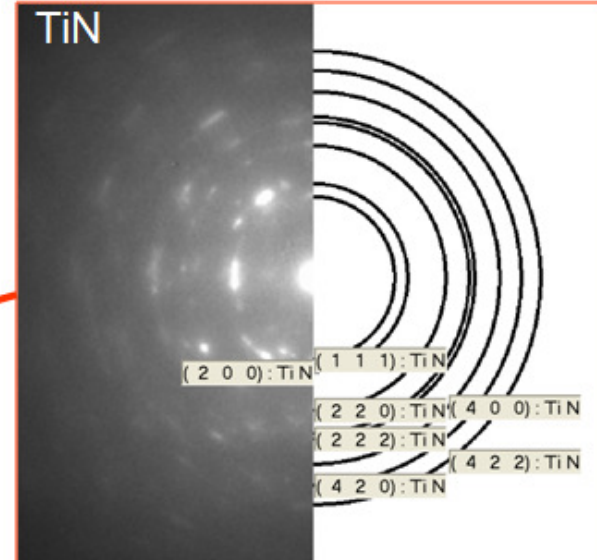
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TEM *BF*



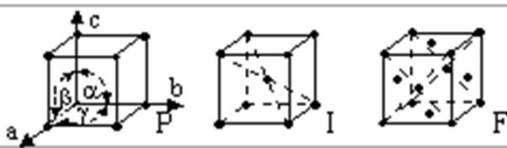
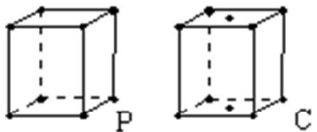
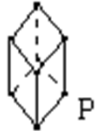
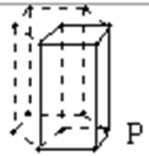
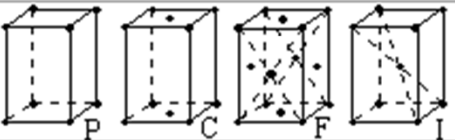
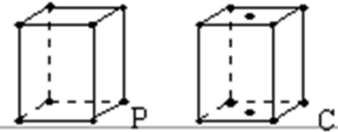
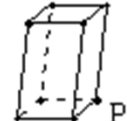
Substrate- austenitic
steel



500 nm





Regularny $\alpha = \beta = \gamma = 90^\circ$ $a = b = c$	23, $m\bar{3}$, $\bar{4}3m, 432,$ $m\bar{3}m$	
Tetragonalny $\alpha = \beta = \gamma = 90^\circ$ $a = b \neq c$	4, $\bar{4}$, $4/m, 422,$ $\bar{4}2m,$ $4mm,$ $4/mmm$	
Trygonalny $\alpha = \beta = \gamma \neq 90^\circ$ $a = b = c$	$3, \bar{3},$ $3m,$ $\bar{3}2,$ $\bar{3}m$	
Heksagonalny $\alpha = \beta = 90^\circ$ $\gamma = 120^\circ$ $a = b \neq c$	$6, \bar{6},$ $622,$ $6/m, \bar{6}m2,$ $6mm,$ $6/mmm$	
Rombowy $\alpha = \beta = \gamma = 90^\circ$ $a \neq b \neq c$	$222,$ $mm,$ mmm	
Jednoskośny $\alpha = \gamma = 90^\circ$ $\beta \neq 90^\circ$ $a \neq b \neq c$	2, $m,$ $2/m$	
Trójskośny $\alpha \neq \beta \neq \gamma \neq 90^\circ$ $a \neq b \neq c$	$1, \bar{1}$	



Miller indices of major crystal planes of the regular system

Bragg's Law:

$$n \lambda = 2 d \sin (\theta)$$

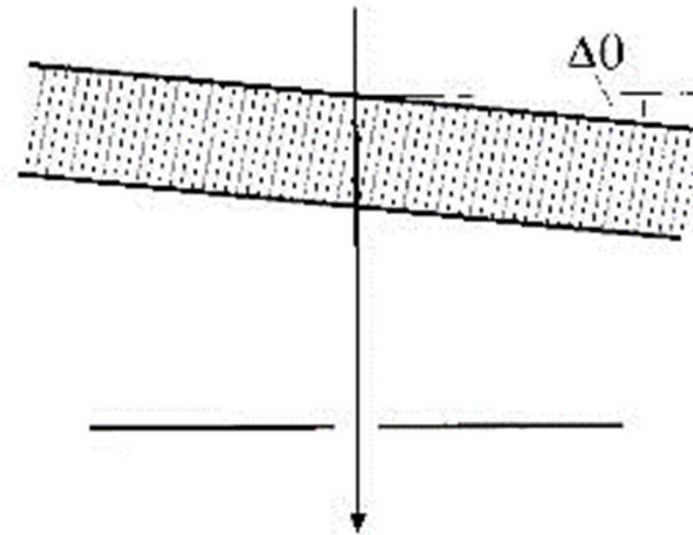
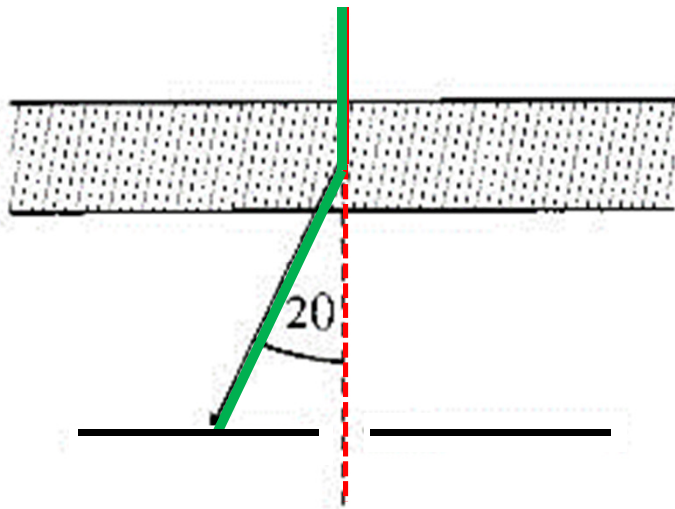
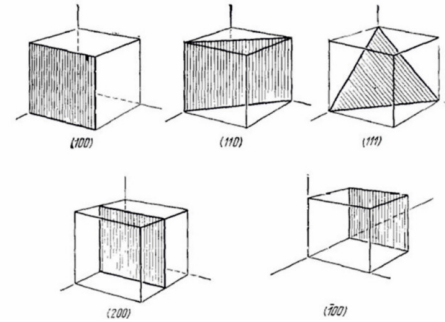
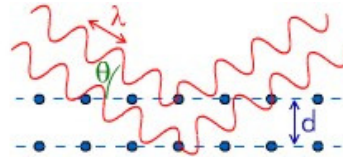
where

λ is the wavelength of the rays

θ is the angle between the incident rays and the surface of the crystal

d is the spacing between layers of atoms

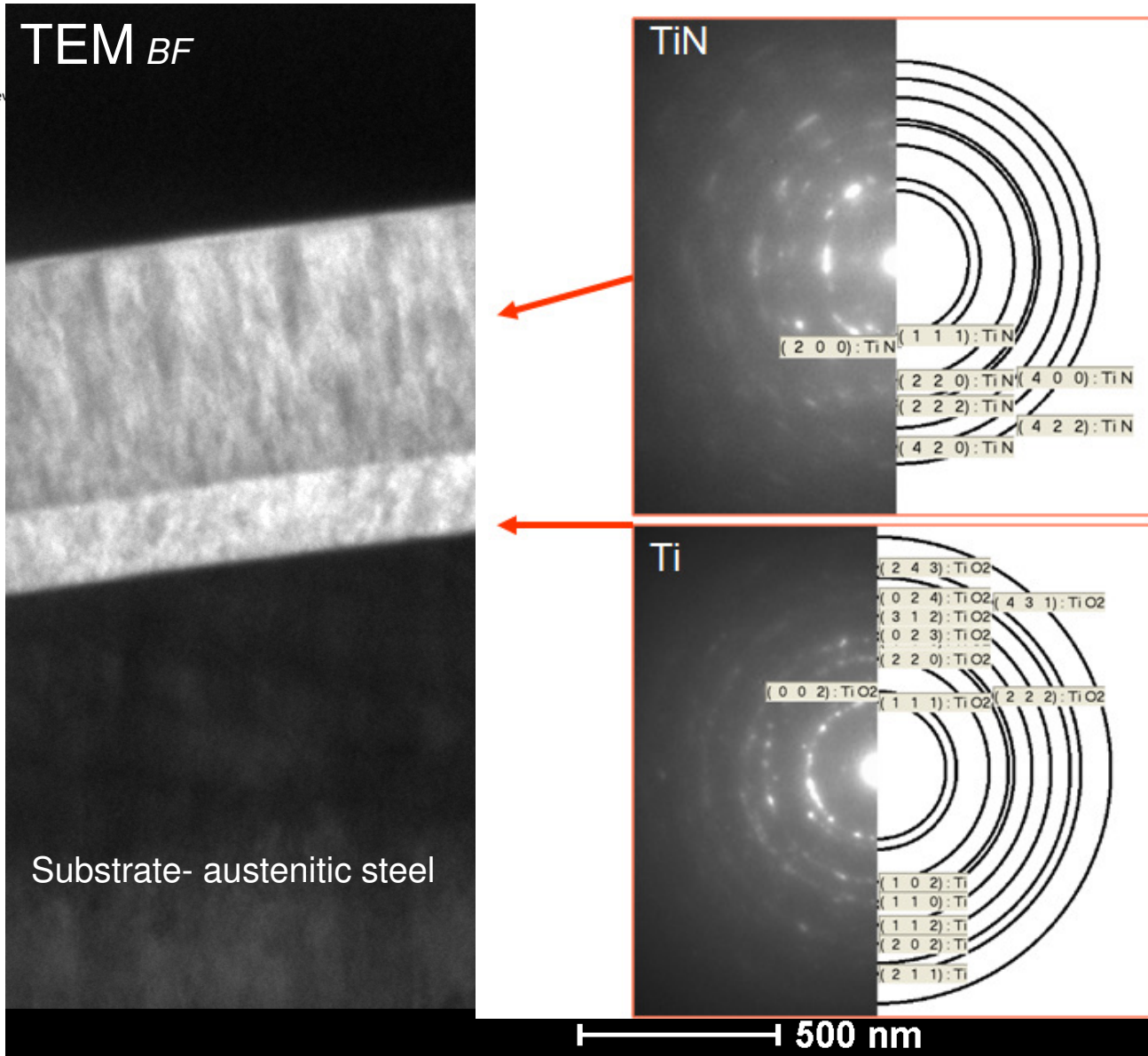
and constructive interference occurs when n is an integer (whole number)



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Single layer TiN coating

Coating after mechanical tests (Ball-on-disc 1N; 2000 cycles)
Microstructure characterization

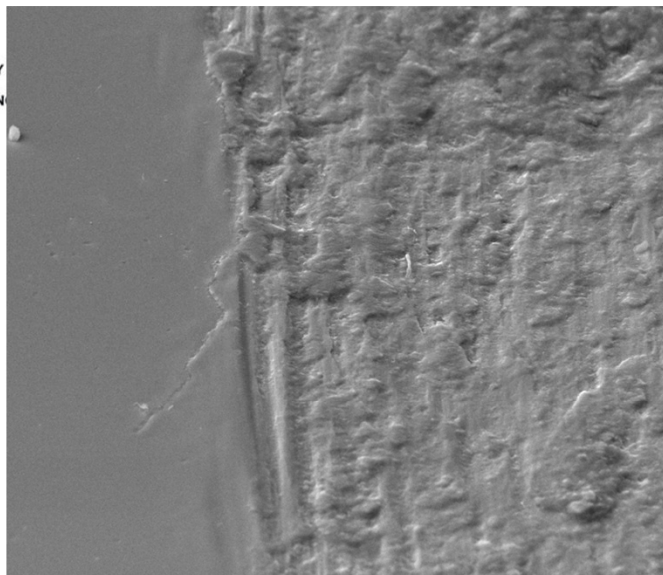
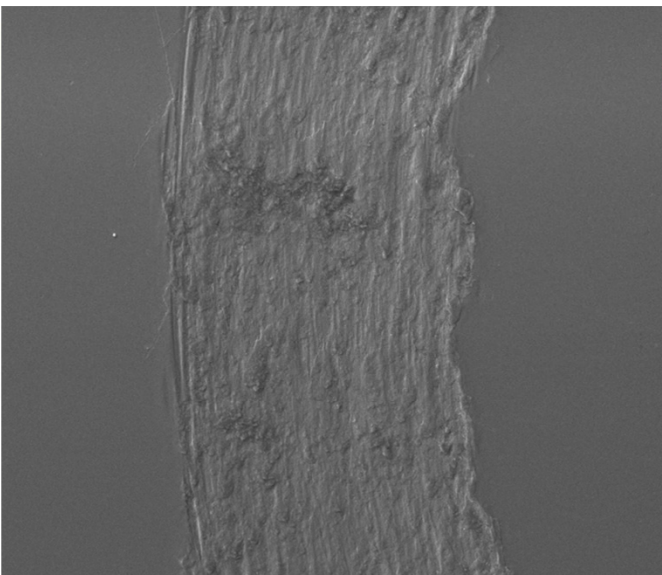
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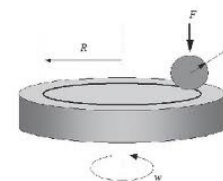
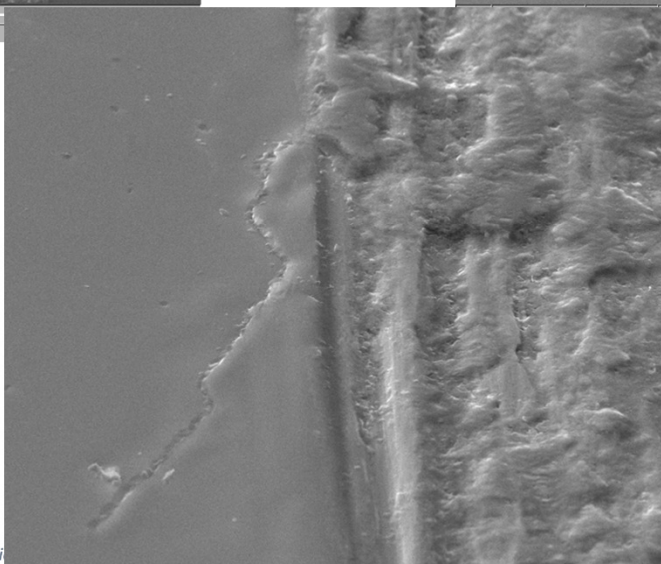


SEM
INSTITUTE OF METALLURGY
AND MATERIALS SCIENCE
POLISH ACADEMY OF SCIENCES



9/9/2011	HV	mag	WD	HFV
3:26:14 PM	30.00 kV	150 x	15.4 mm	901 μ m

mag	WD	HFV	50 μ m
100 x	15.0 mm	225 μ m	p4J3.4



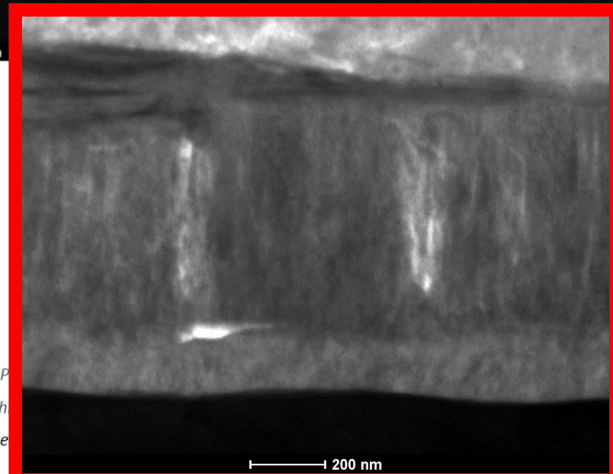
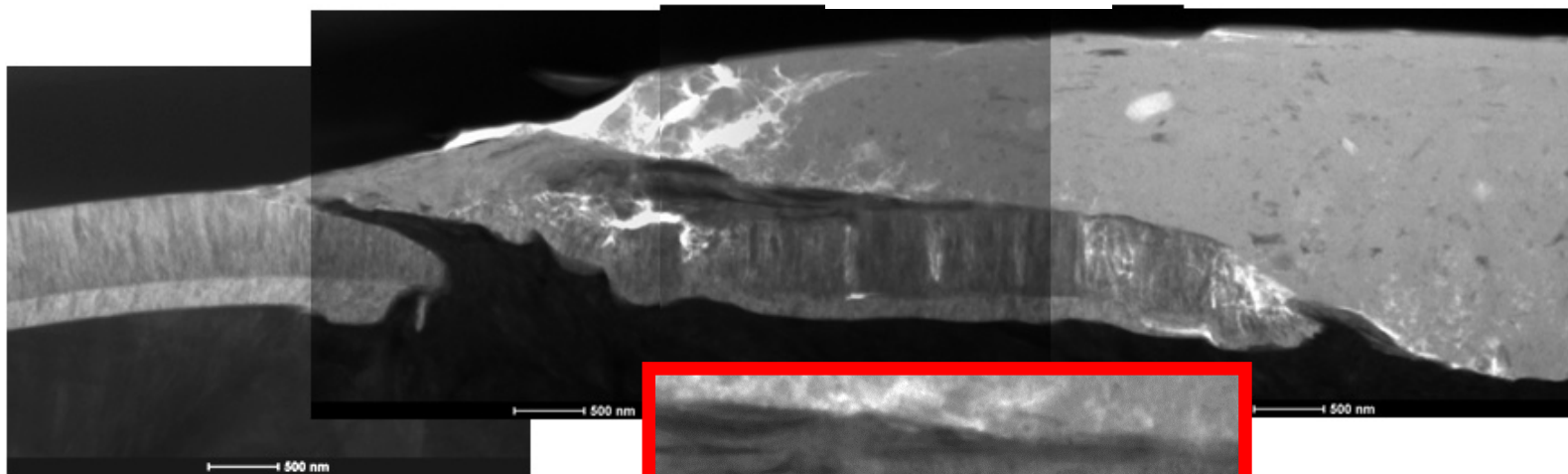
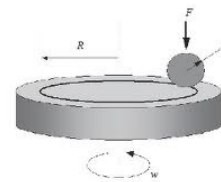
Internati

ction

9/9/2011	HV	mag	WD	HFV	40 μ m
3:34:15 PM	30.00 kV	1300 x	15.0 mm	104 μ m	p4J3.4

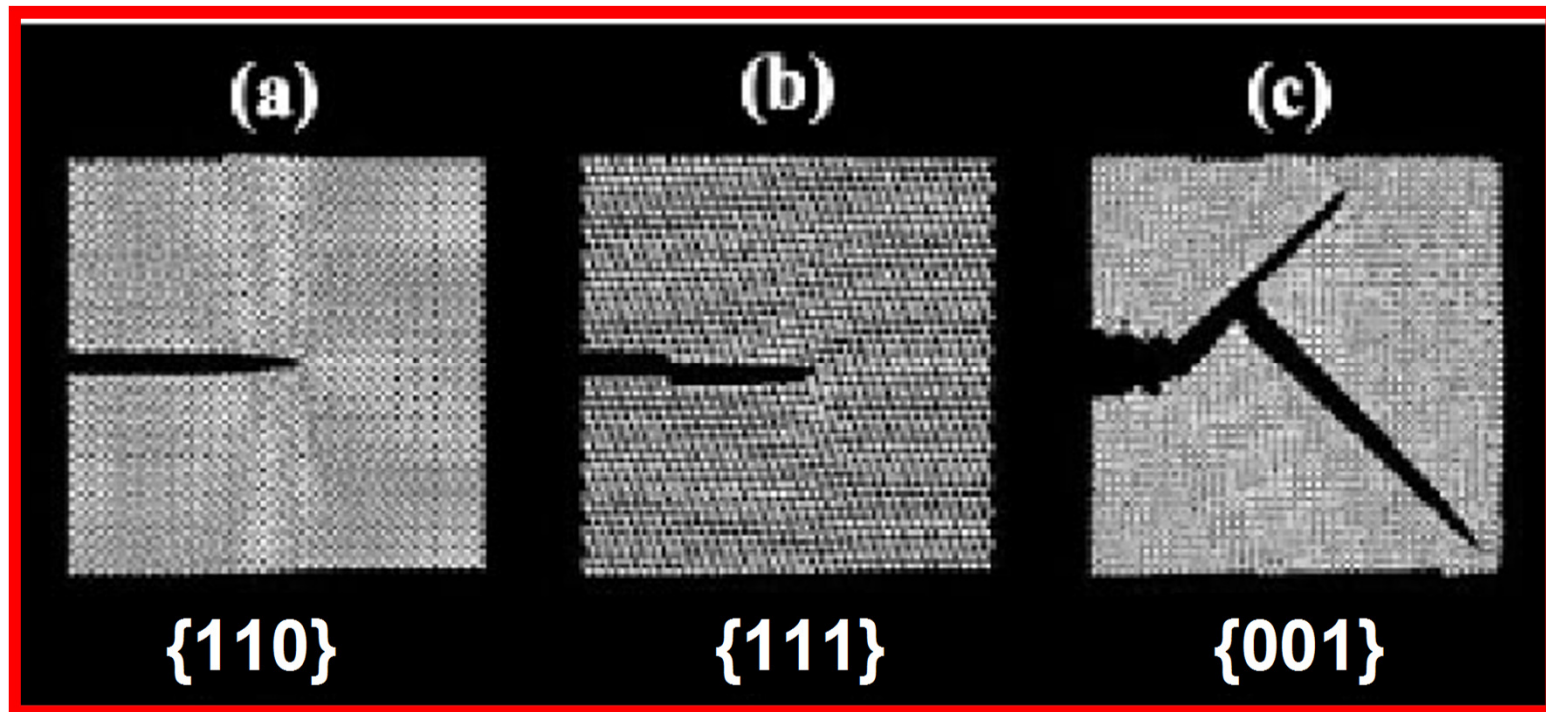


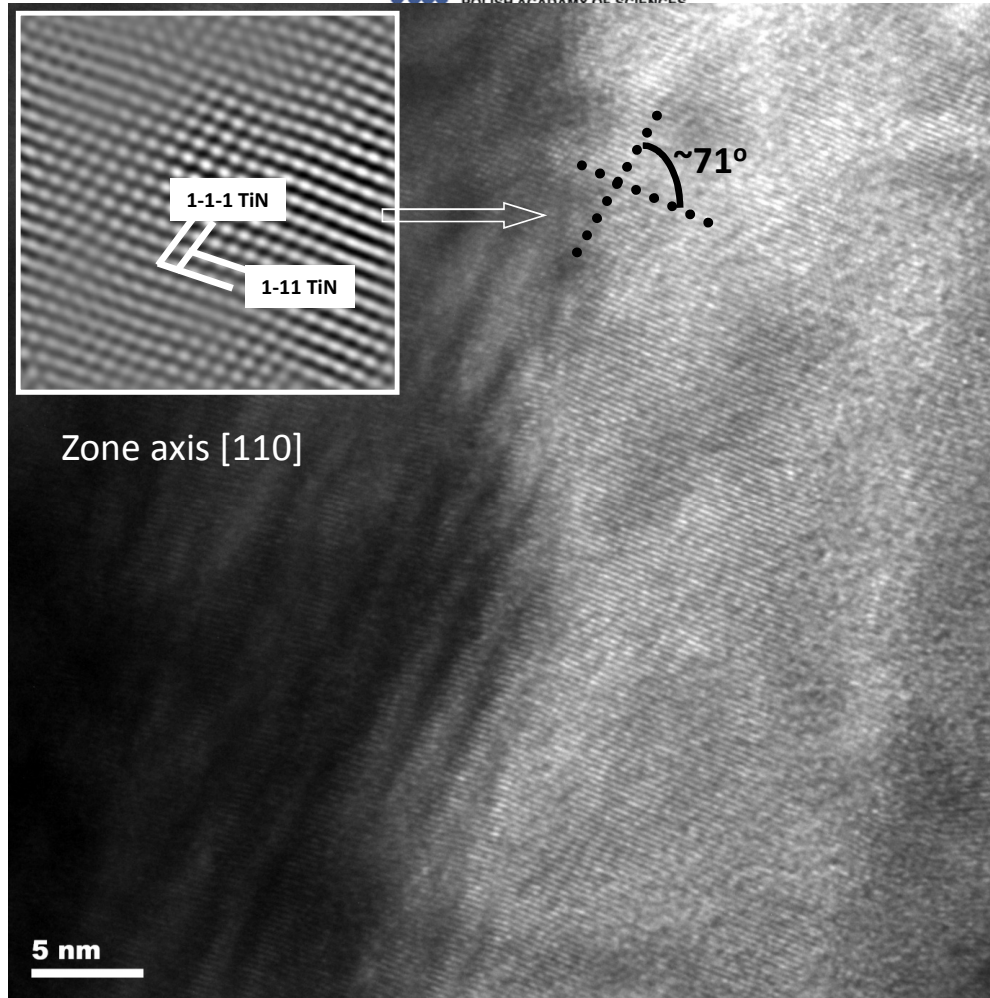
TEM *BF*; *Coating after mechanical tests*





Atomic structures images of brittle cracking along particular crystallographic planes:





TiN

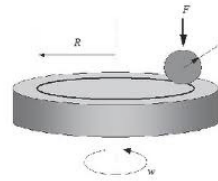
(1 1 1)

(-1 1 1)

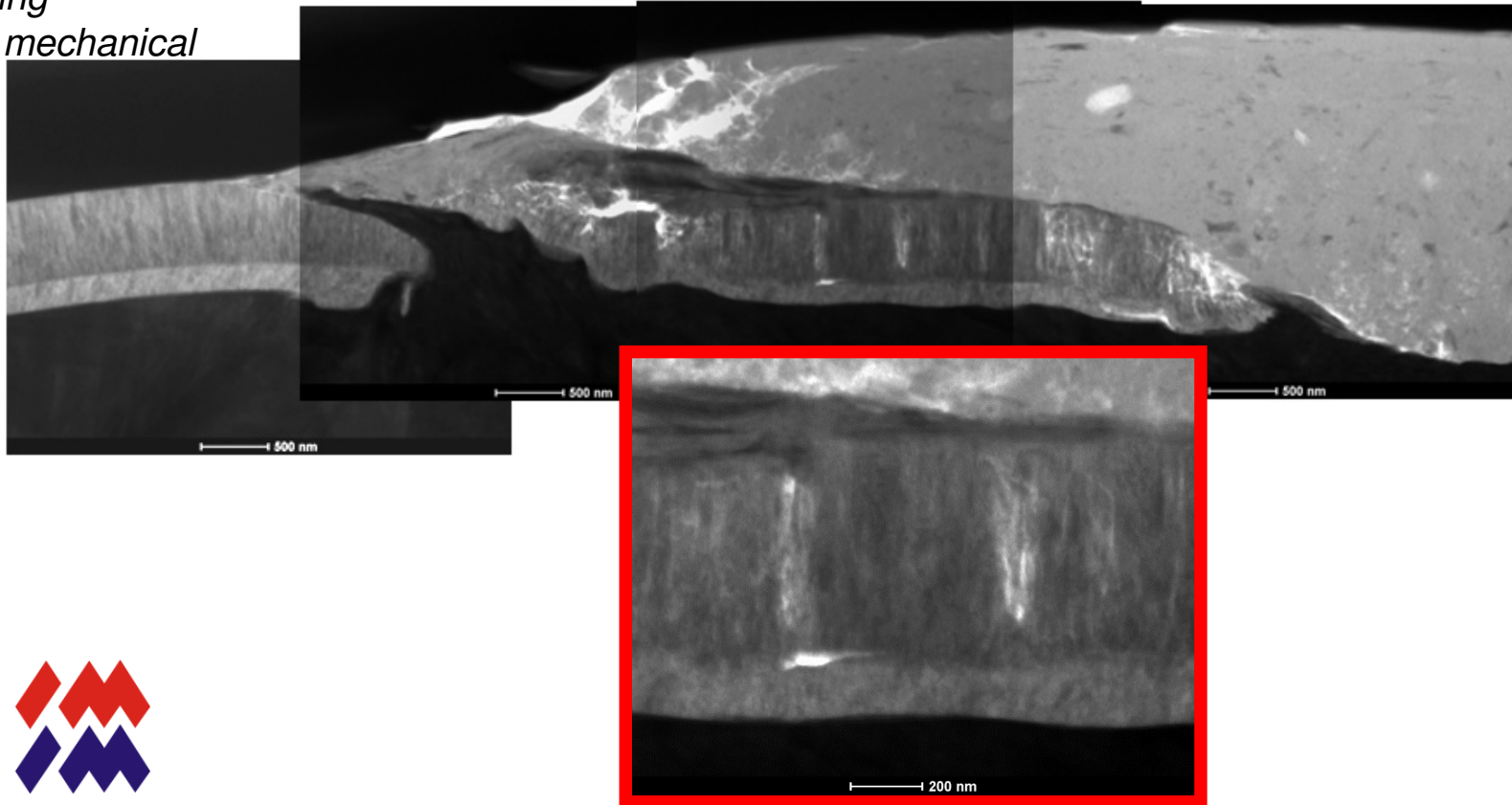
(1 -1 1)

(-1 -1 1)





TEM *BF*; Coating after mechanical tests



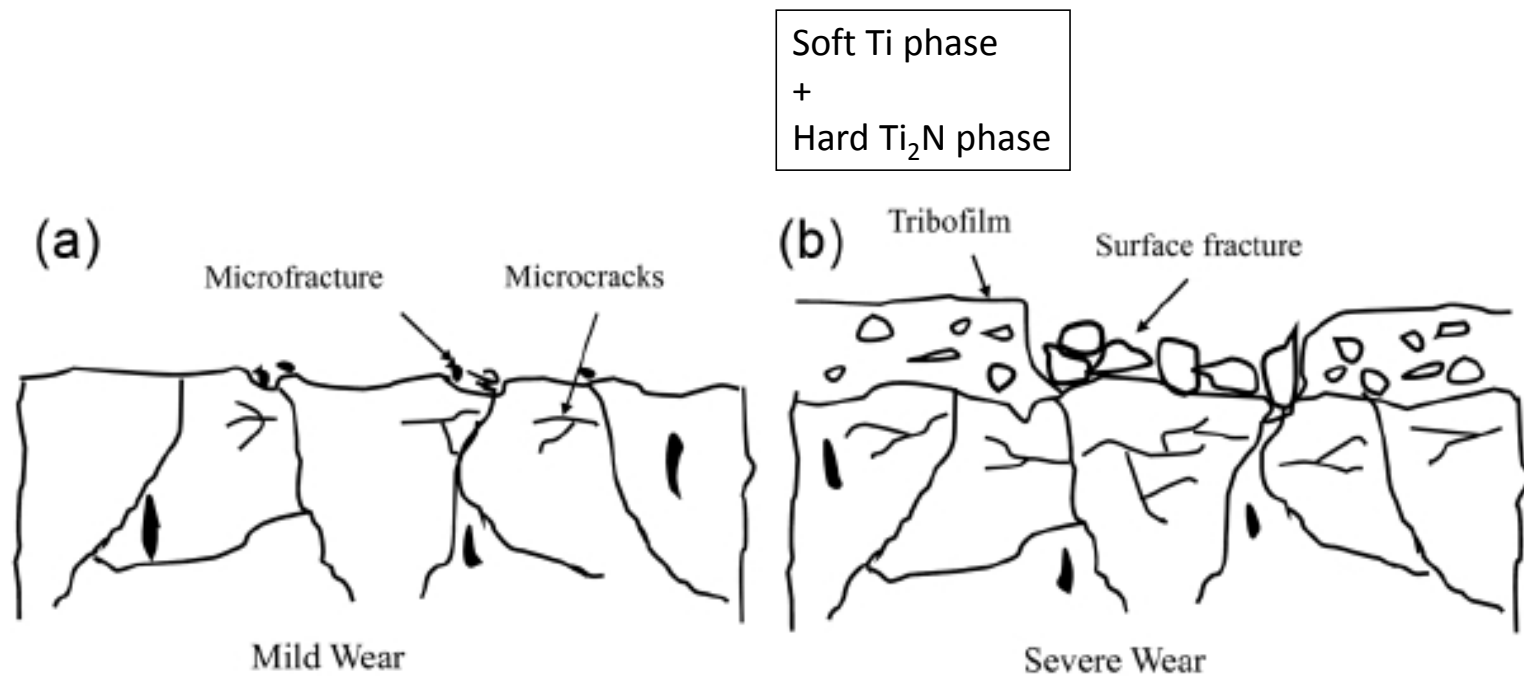
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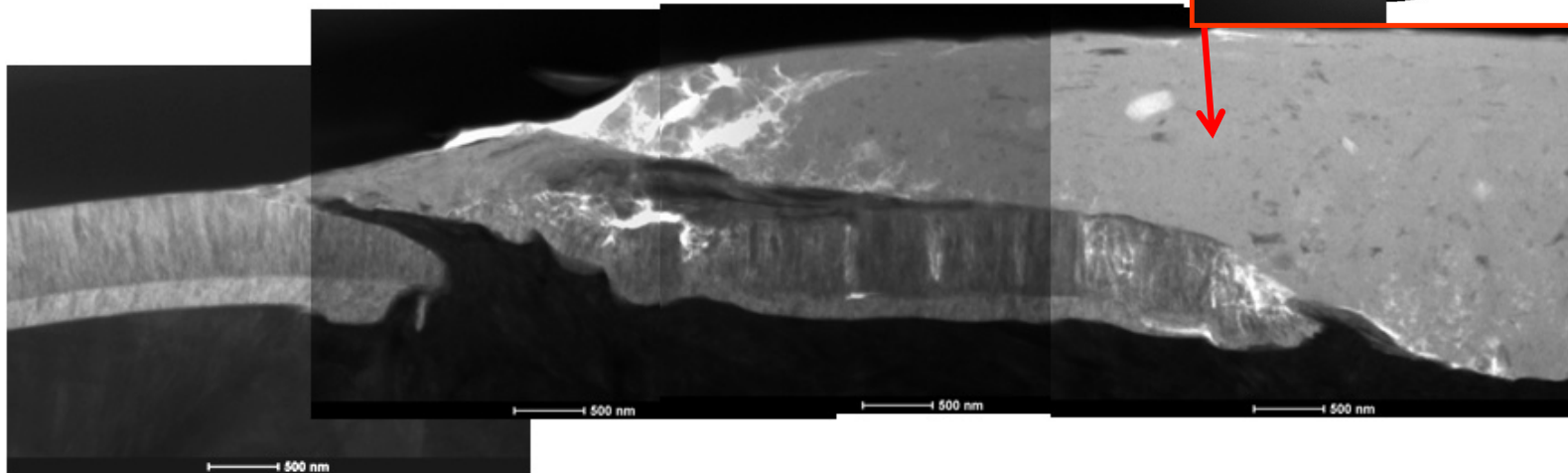
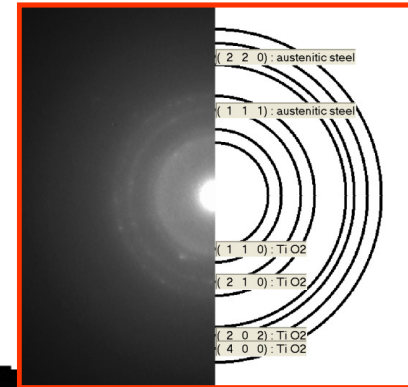
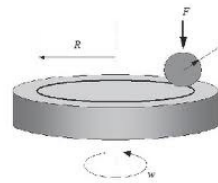


Microstructural characterization of the coatings by TEM after the wear test

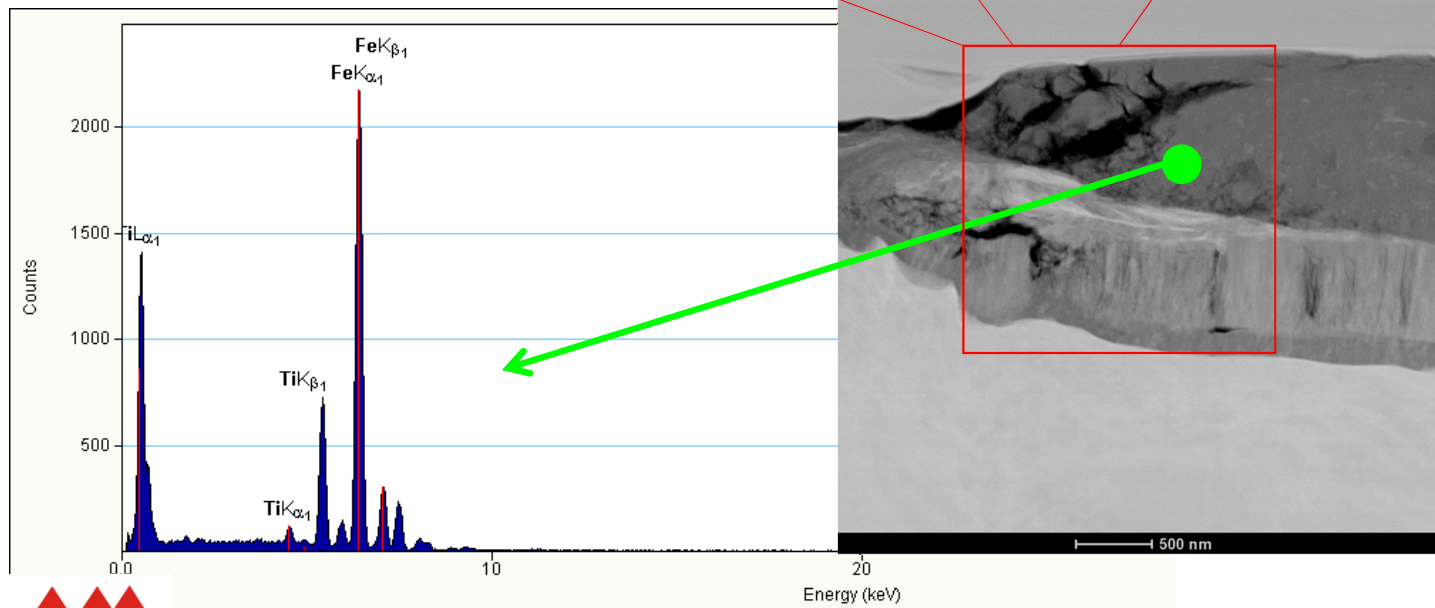
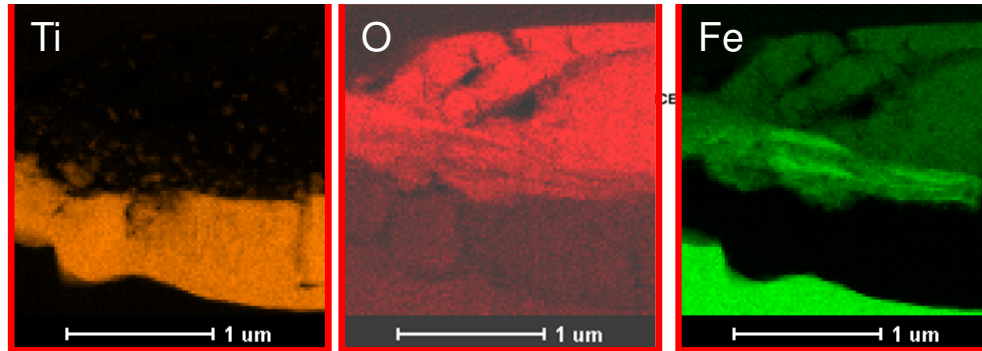




TEM *BF*; *Coating after mechanical tests*

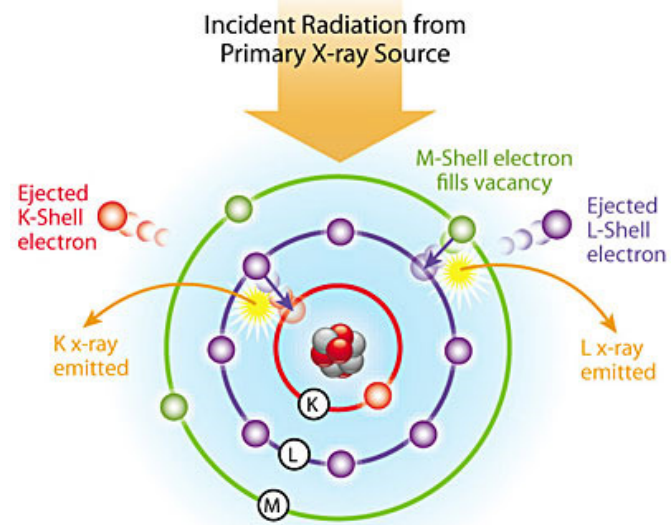
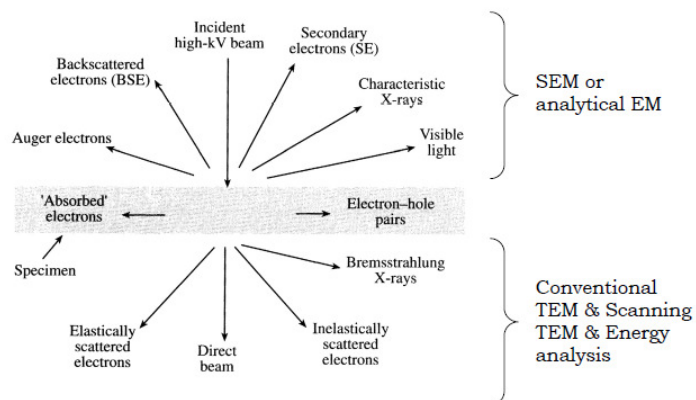


Qualitative
chemical
analysis
EDS



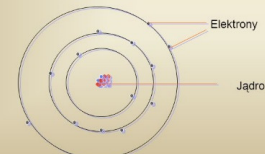


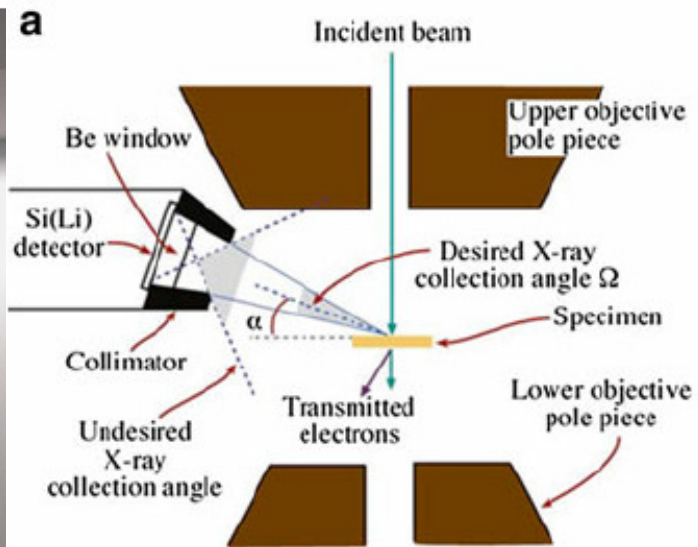
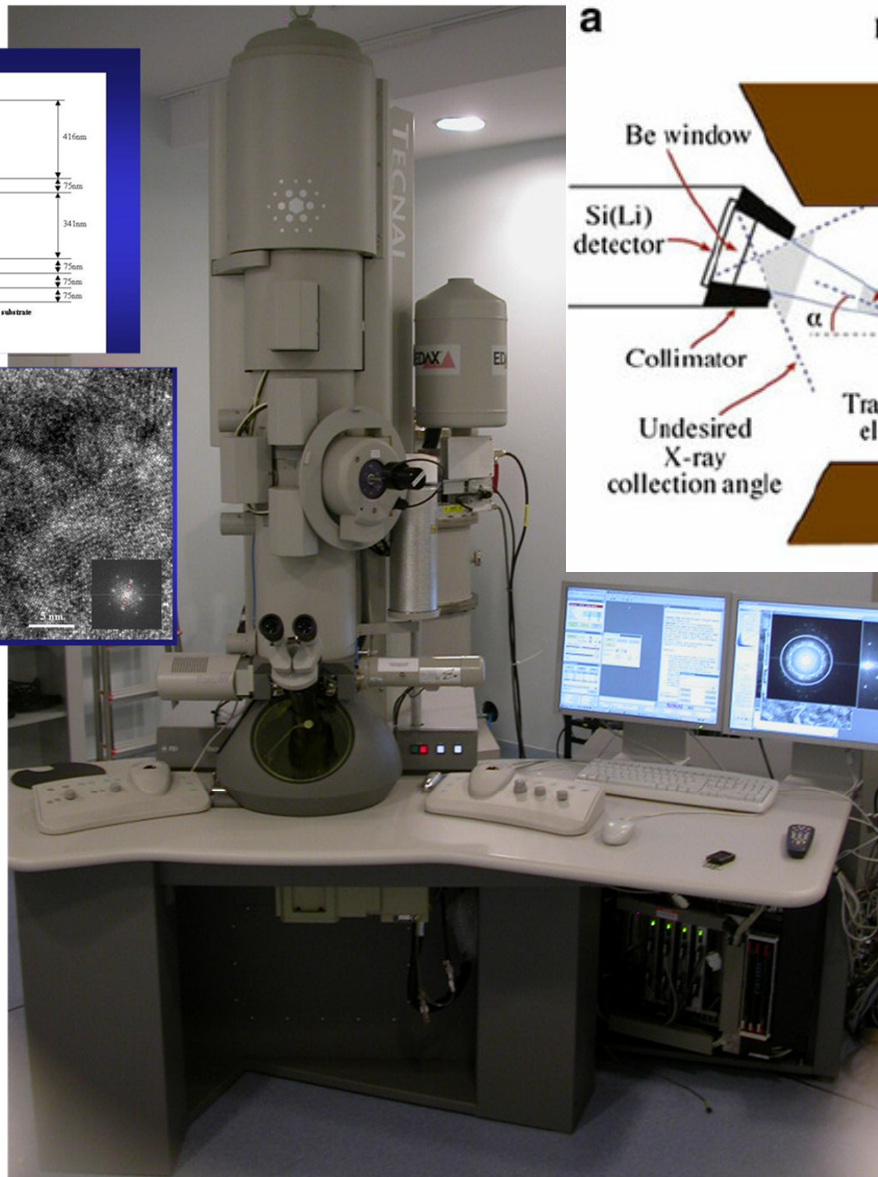
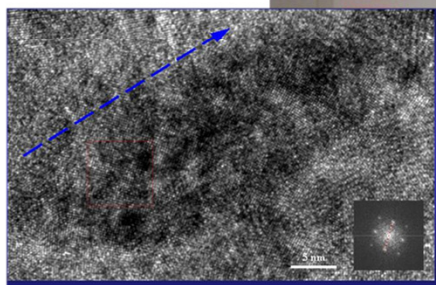
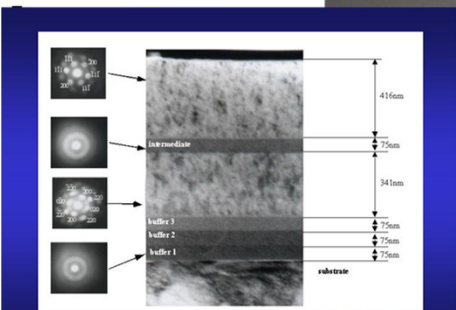
Interaction of Electron with Samples

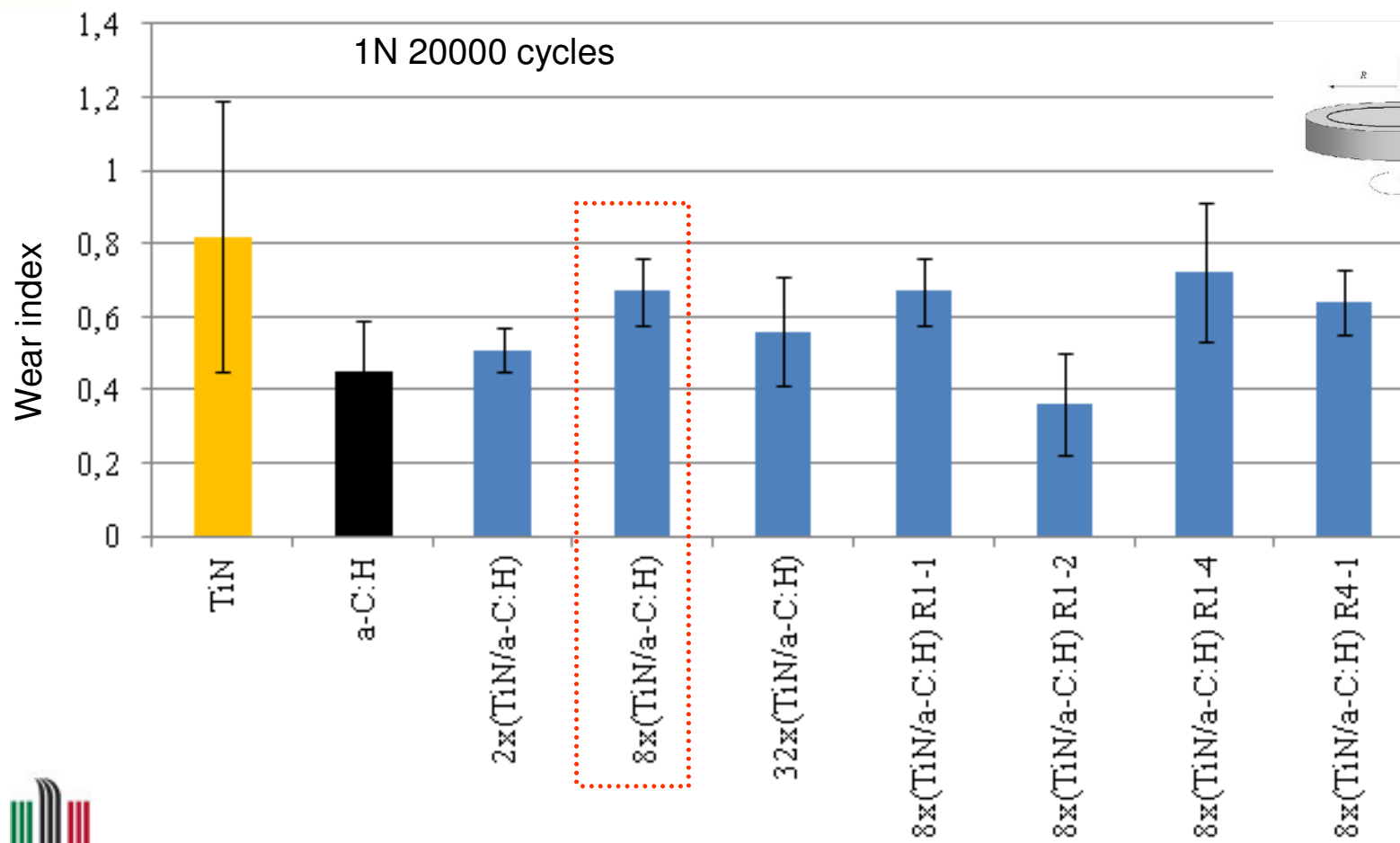


Atom Bohra

Model budowy atomu Bohra – model atomu wodoru autorstwa Nielsa Bohra. Bohr przyjął wprowadzony przez Ernesta Rutherforda model atomu, według tego modelu elektron krąży wokół jądra jako naładowany punkt materialny, przyciągany przez jądro siłami elektrostatycznymi. Przez analogię do ruchu planet wokół Słońca model ten nazwano "modelem planetarnym atomu".









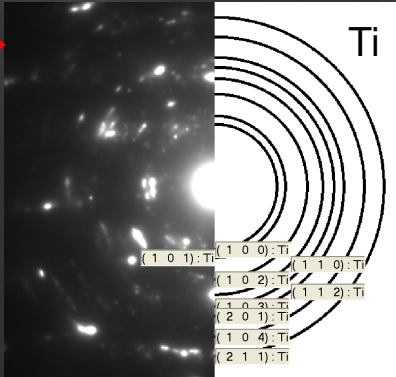
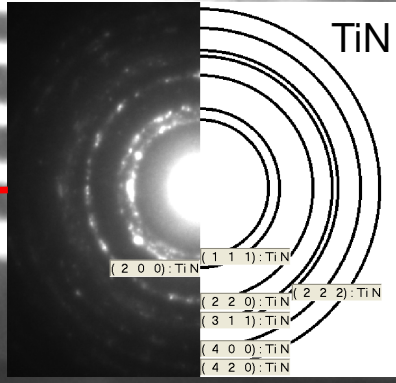
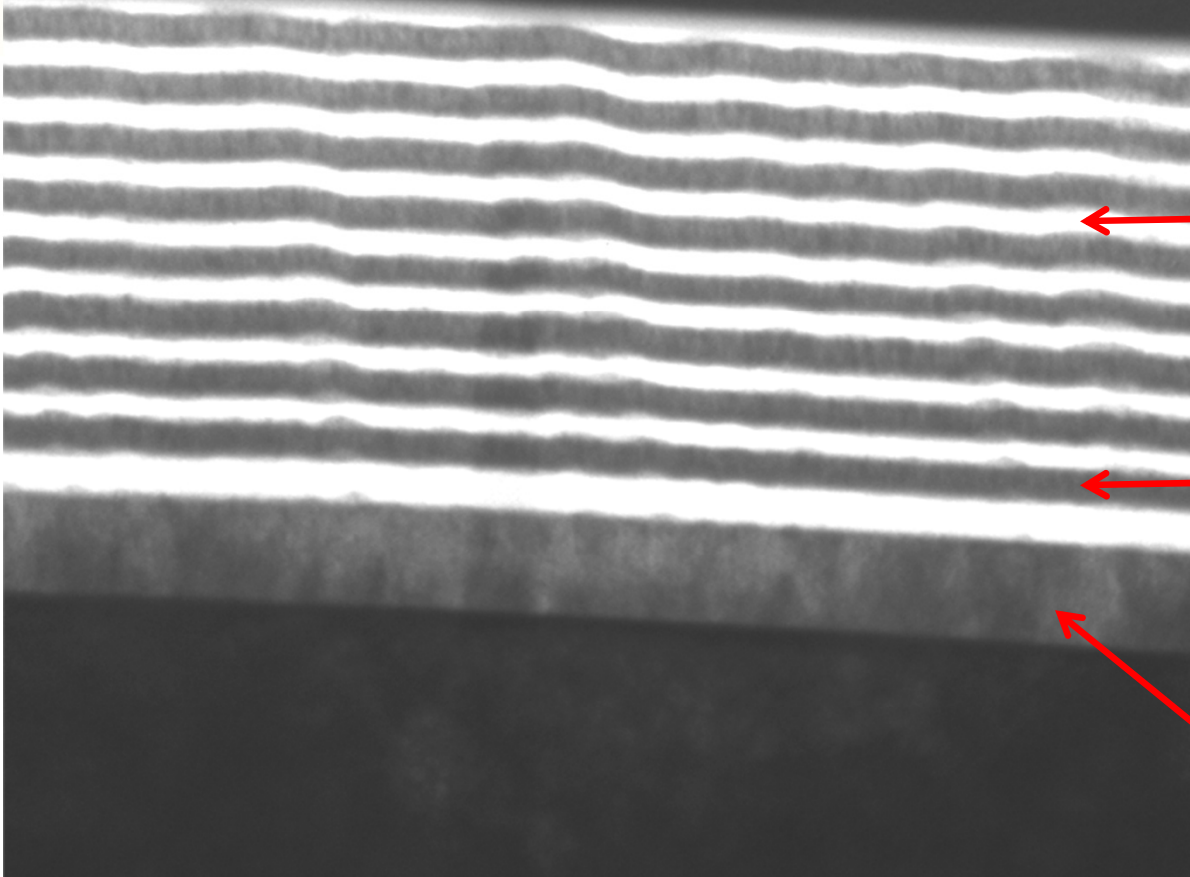
Multilayer 8x TiN/Ti/a-C:H coating

As deposited coating (Before mechanical tests)
Microstructure characterization



TEM *BF* (as-deposited coating)

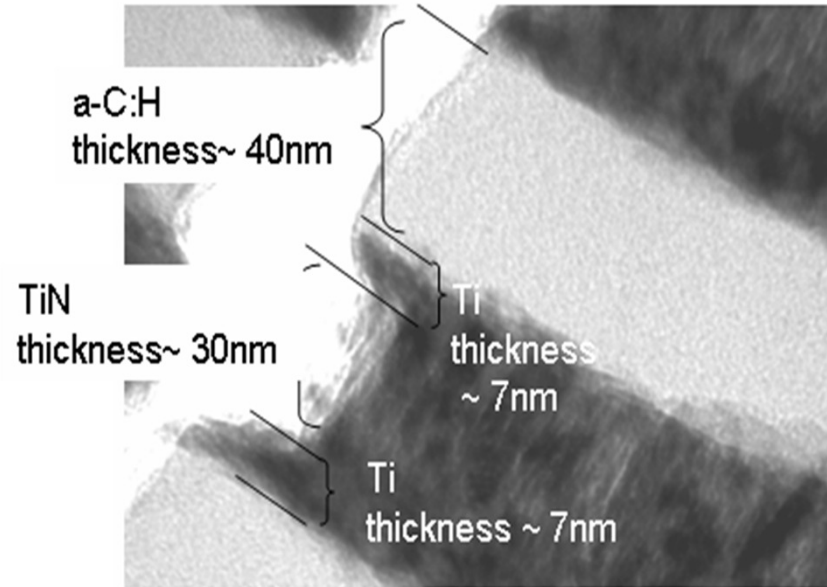
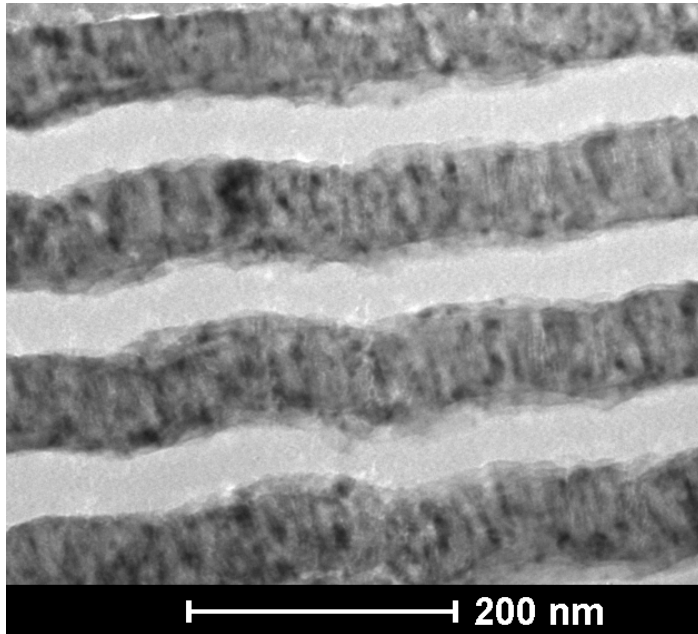
a-C:H



Substrate- austenite steel



500 nm



100 nm



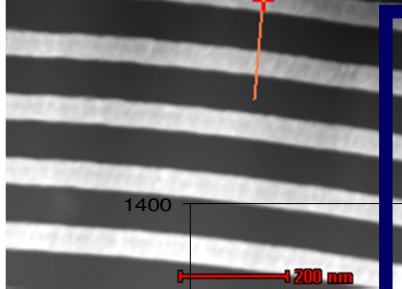
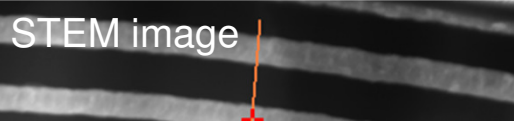
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EU
FUNDING
KNOWLEDGE



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AN
POL

correspond to Ti

European Union
European Social Fund

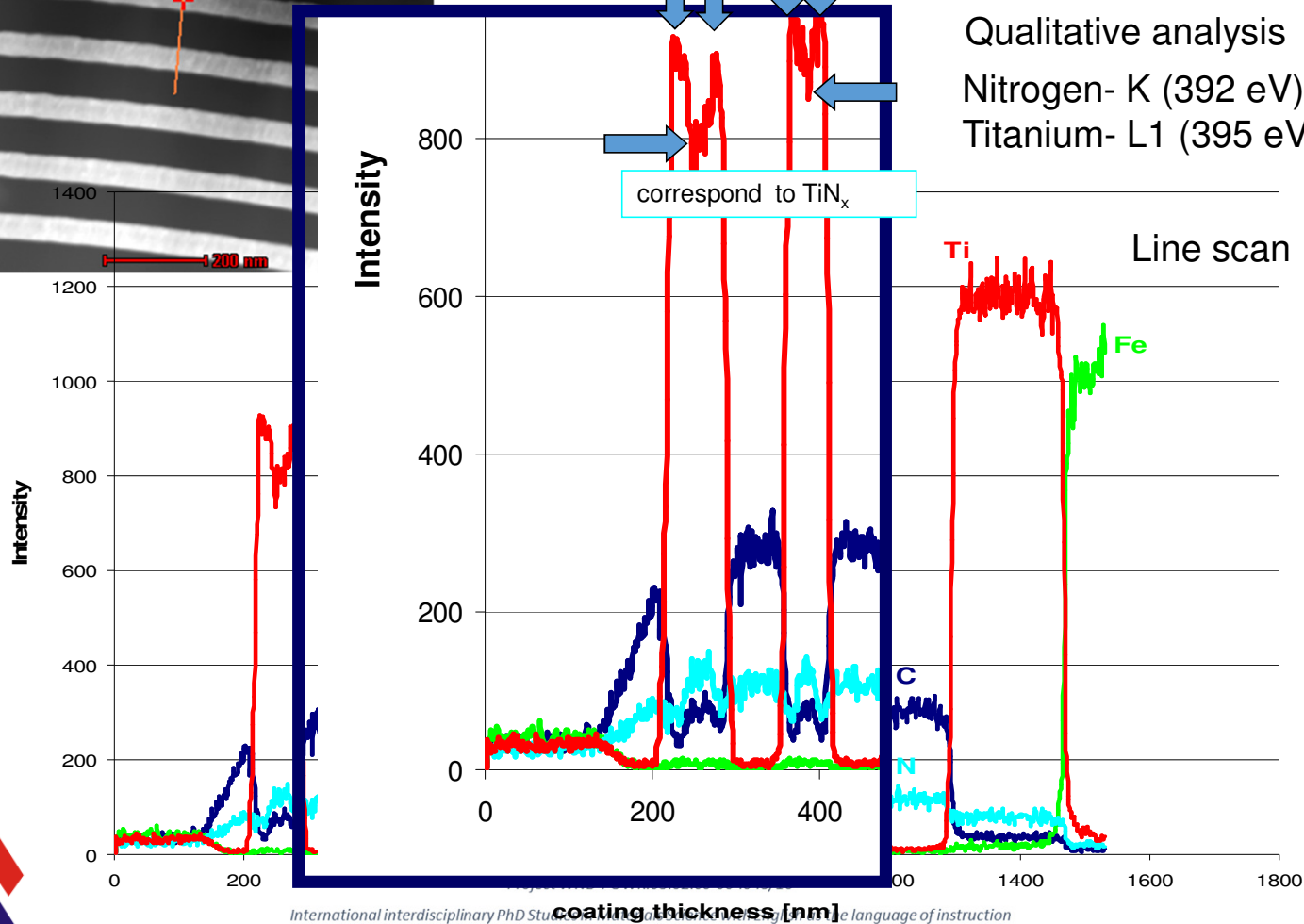


EDS

Qualitative analysis

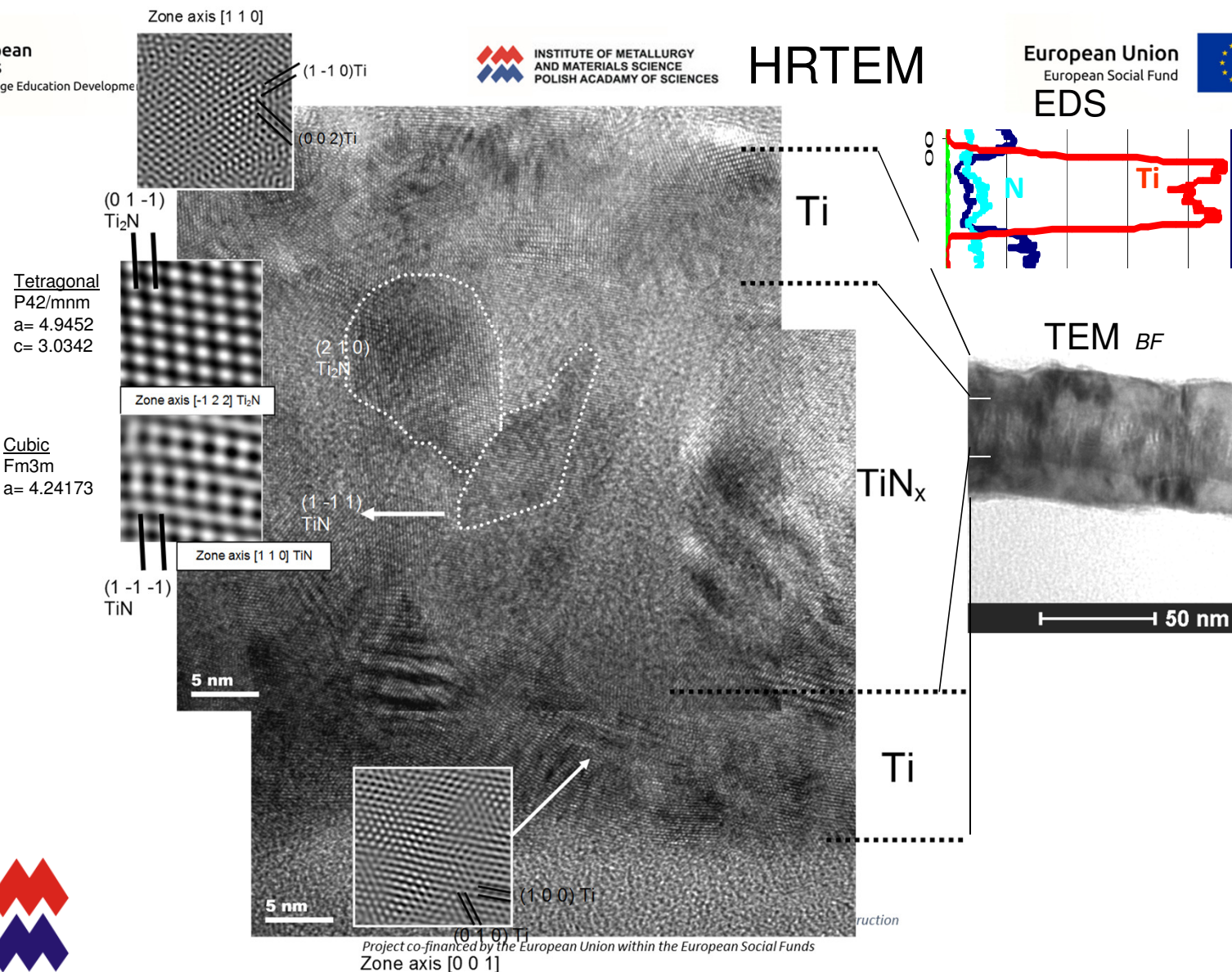
Nitrogen- K (392 eV)

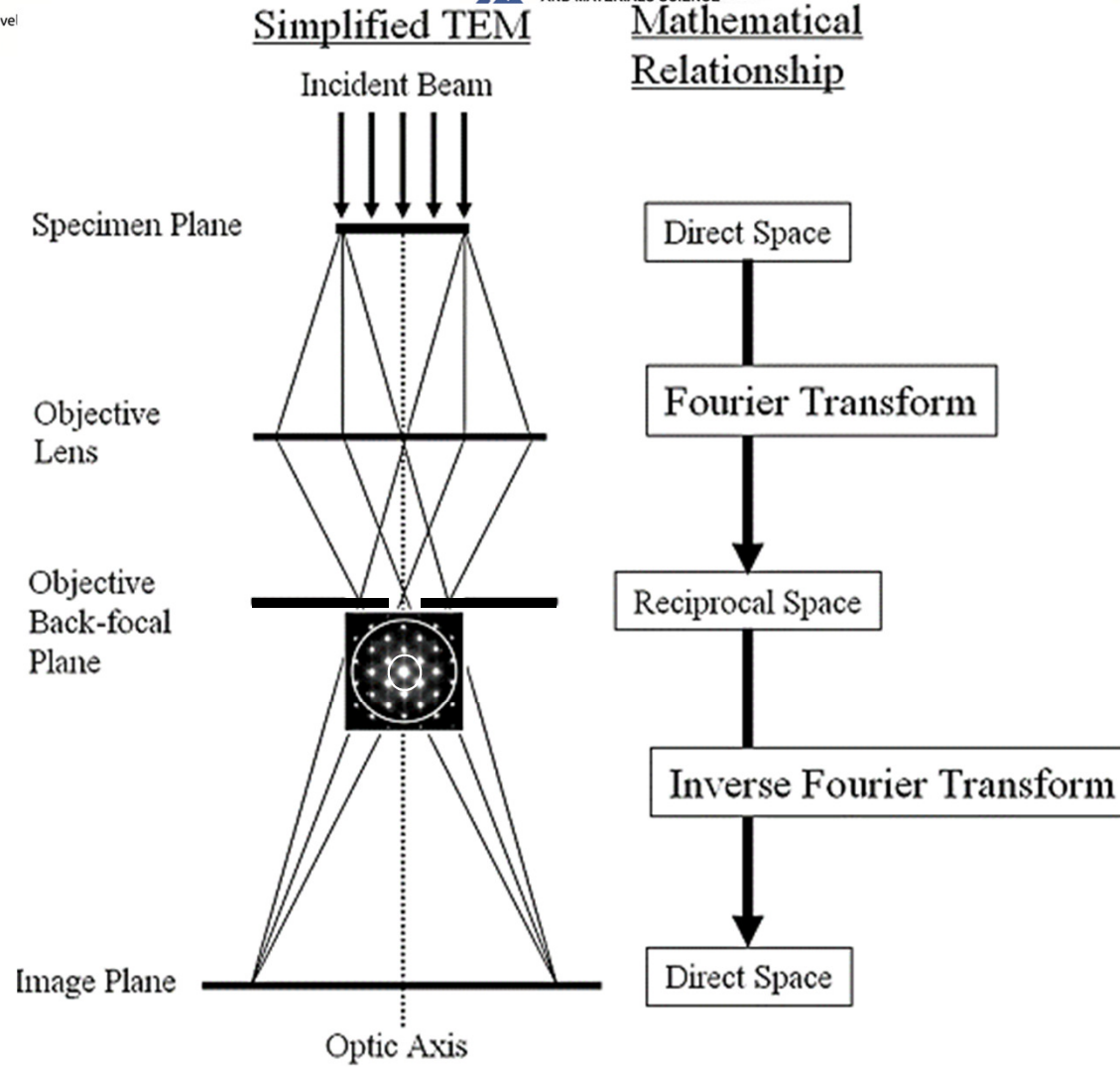
Titanium- L1 (395 eV)



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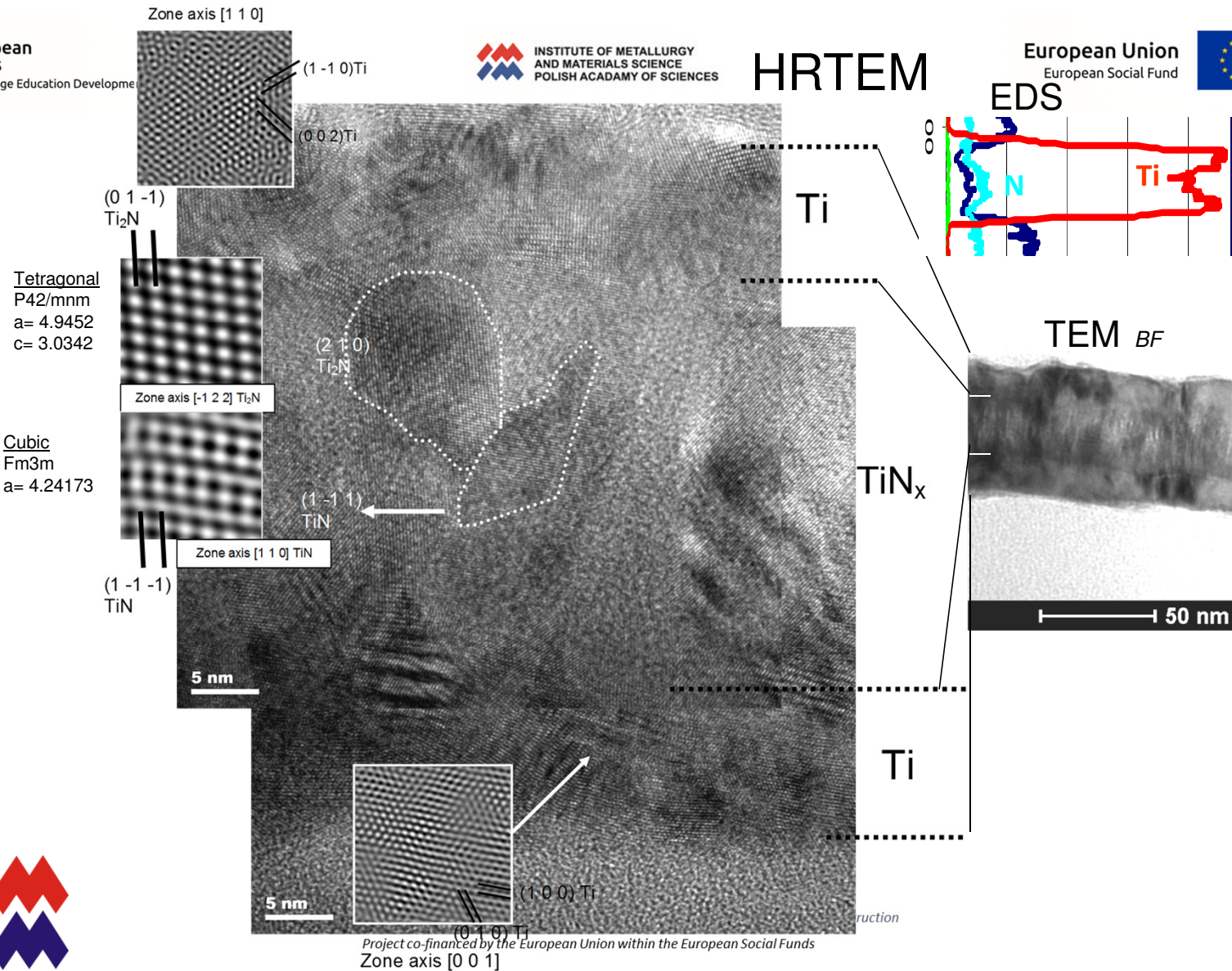


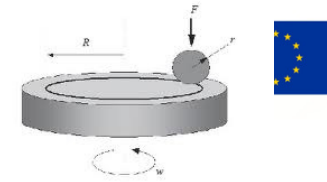




HRTEM

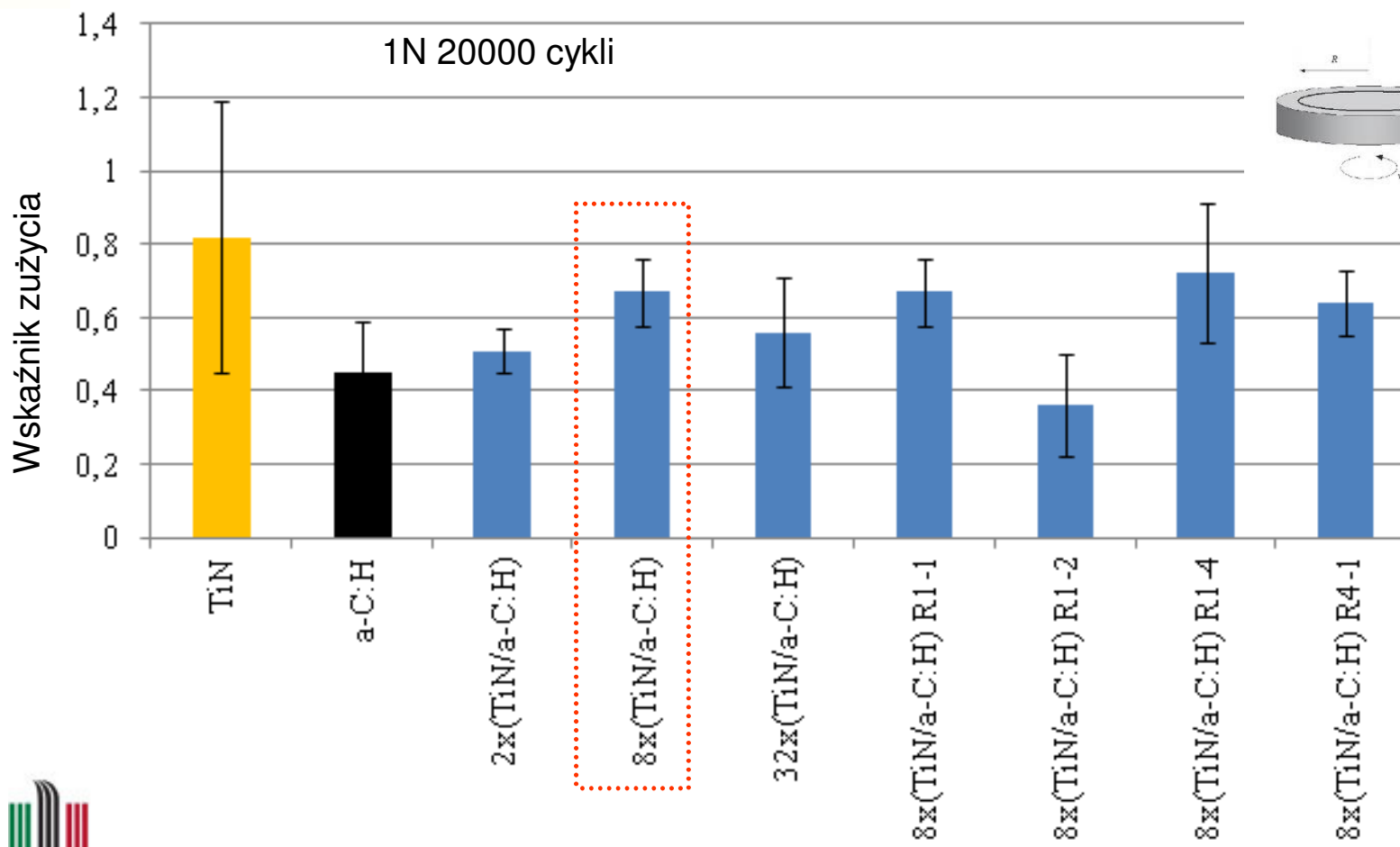
EDS

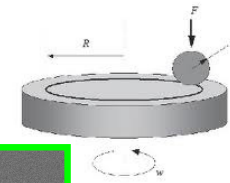




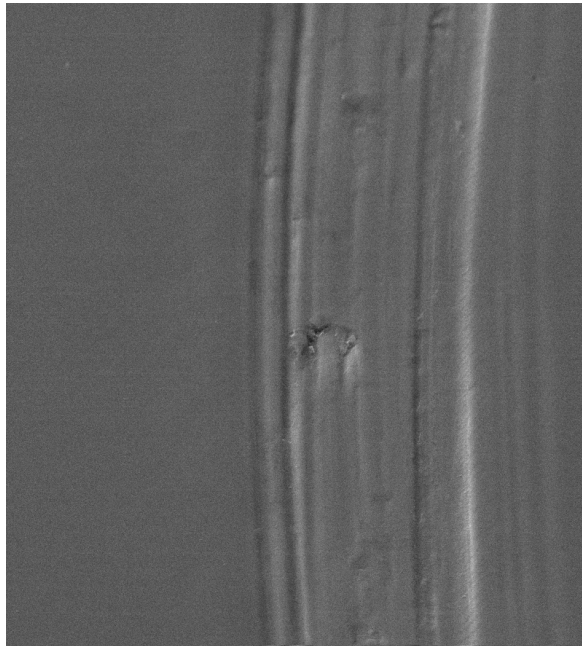
Multilayer 8x TiN/Ti/a-C:H coating

Coating after mechanical tests (Ball-on-disc 1N; 2000 cycles)
Microstructure characterization

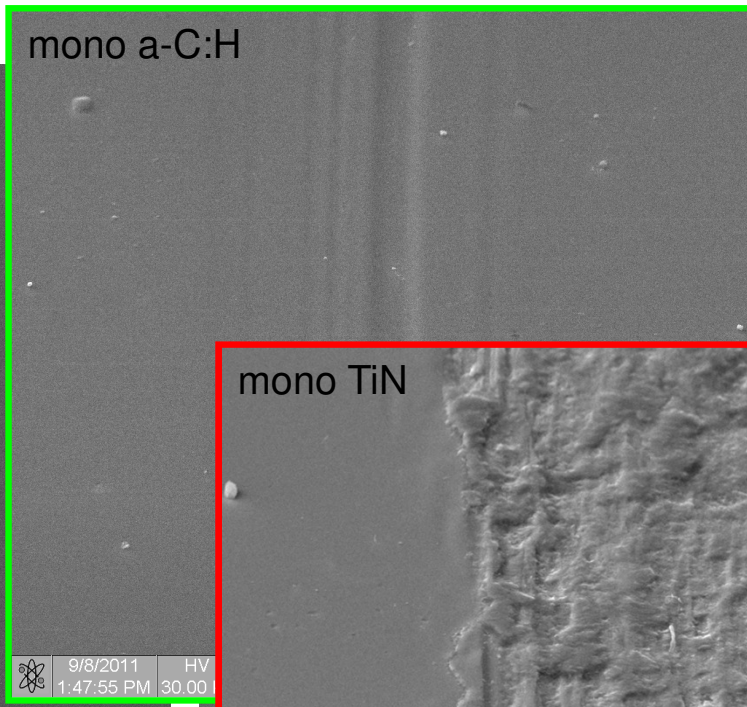




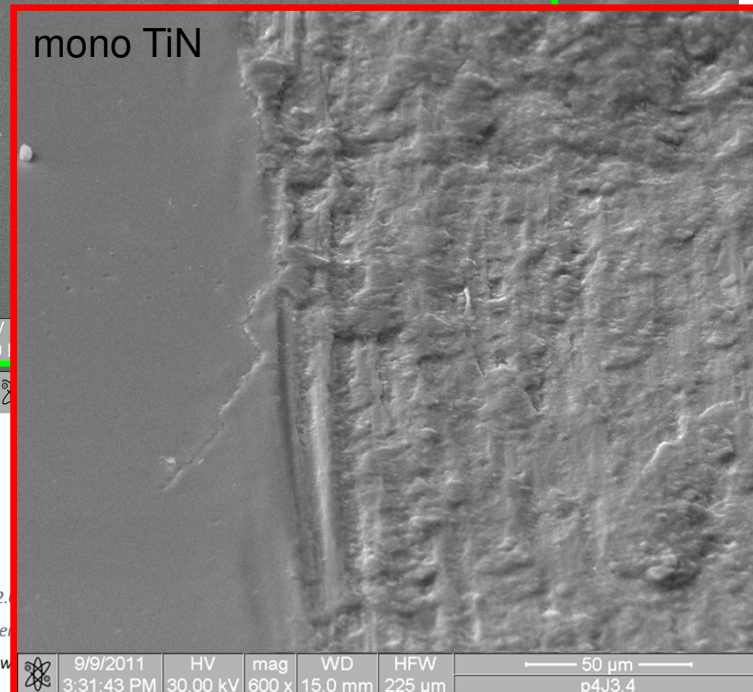
8x TiN/Ti/a-C:H (1:1)



mono a-C:H



mono TiN



	9/15/2011 2:50:21 PM	HV 30.00 kV	mag 600 x	WD 14.9 mm	HPW 225 μm	50 μm p4J3.4
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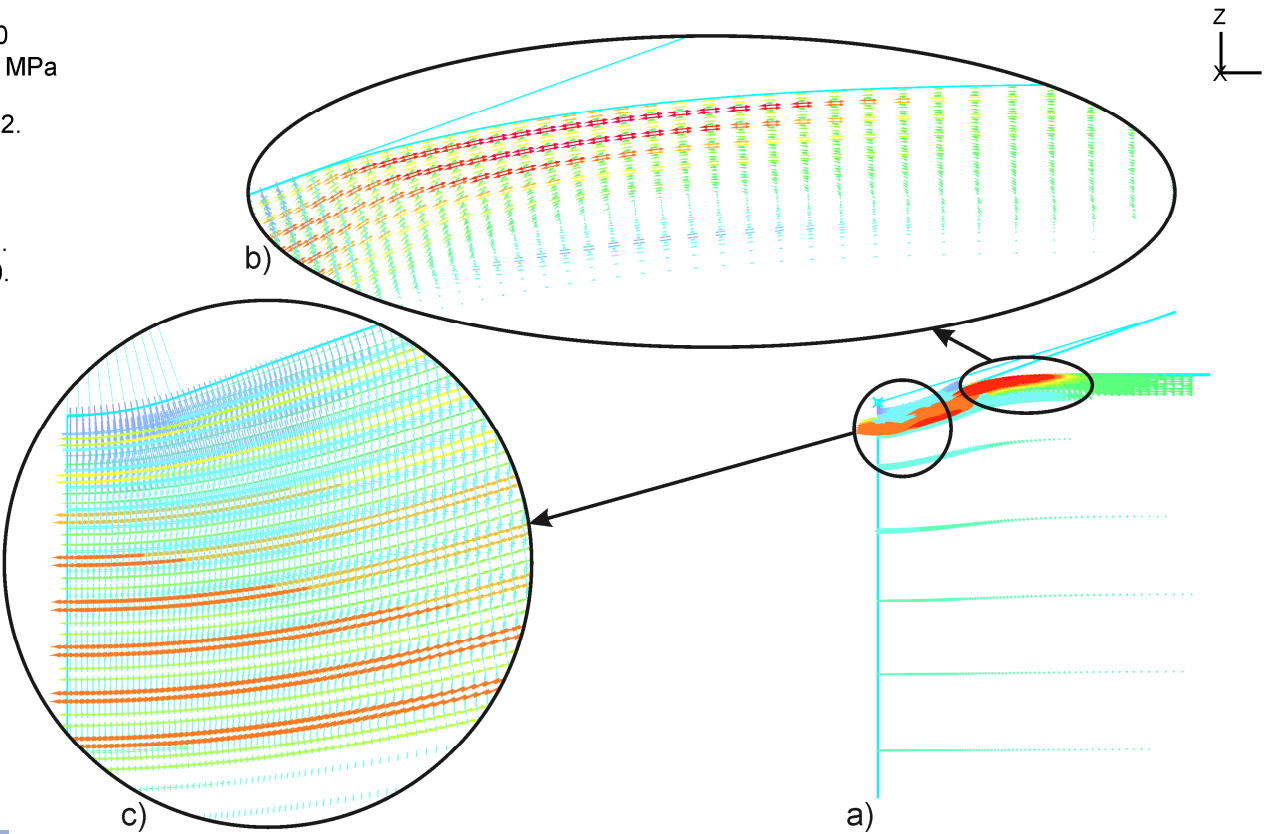
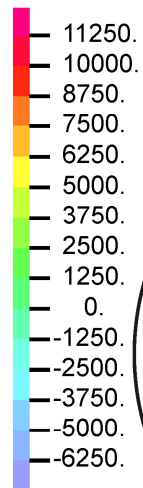
	9/8/2011 1:47:55 PM	HV 30.00 kV
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	9/9/2011 3:31:43 PM	HV 30.00 kV	mag 600 x	WD 15.0 mm	HPW 225 μm	50 μm p4J3.4
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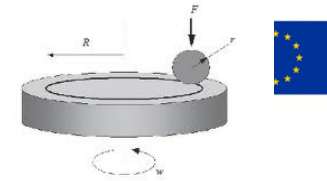
A TIME 30.0
D STRESS, MPa
I + -
N 10572.
A ↑ ↓



Project WND-POWR.03.02.00-00-1043/16

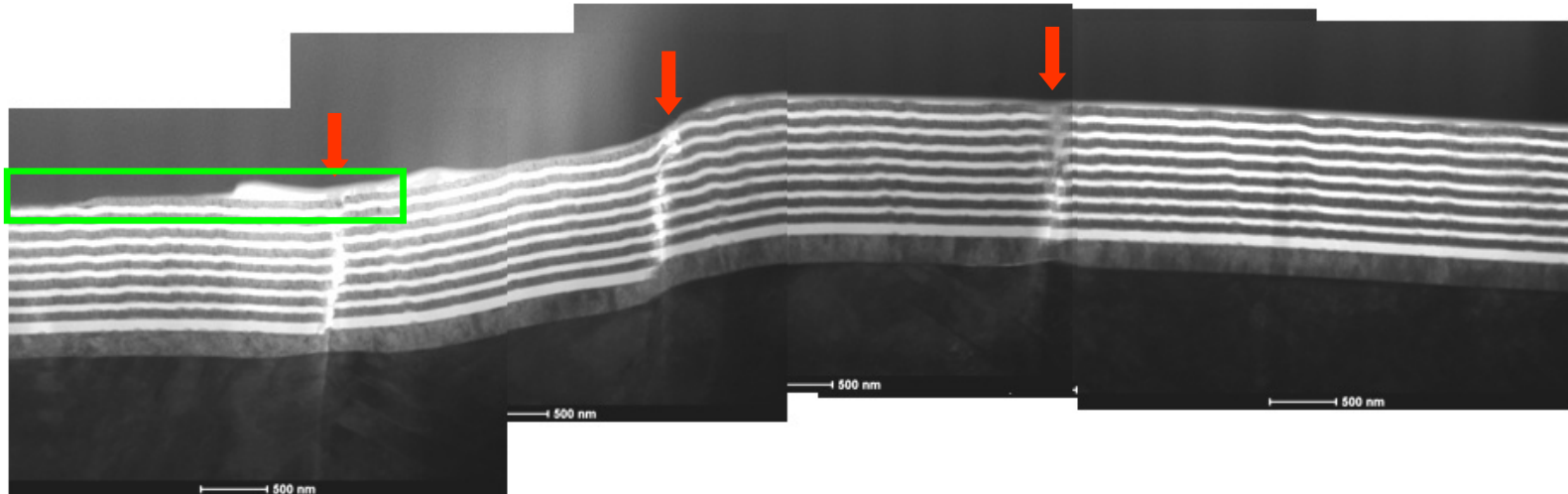
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TEM *BF*; *Coating after mechanical tests*

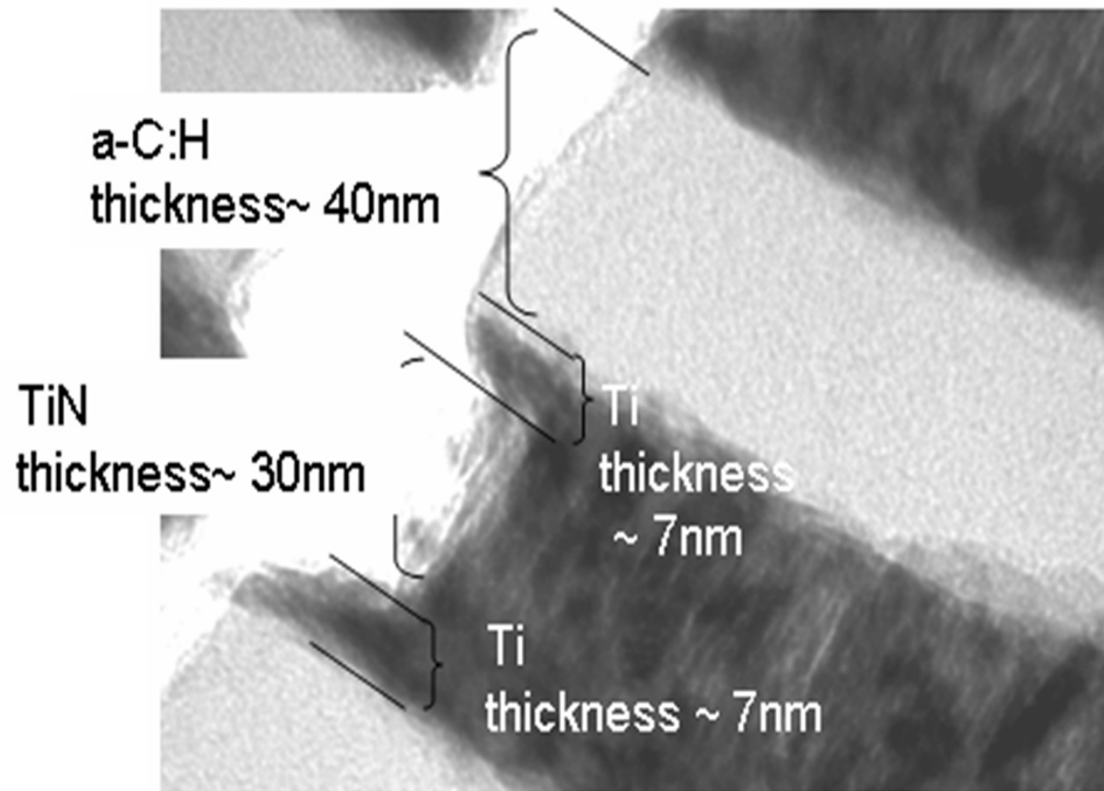
- Wear mechanisms:
- 1) by cracking
 - 2) by layer by layer remove + tribo-film formation





TEM_{BF}

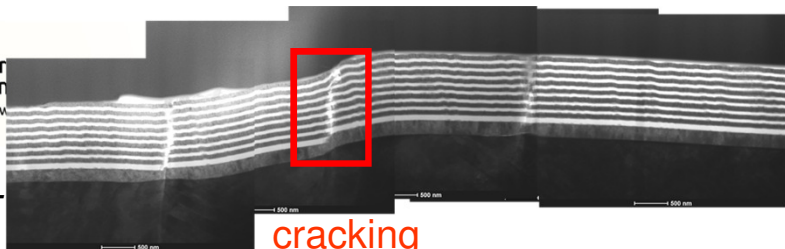
cracking



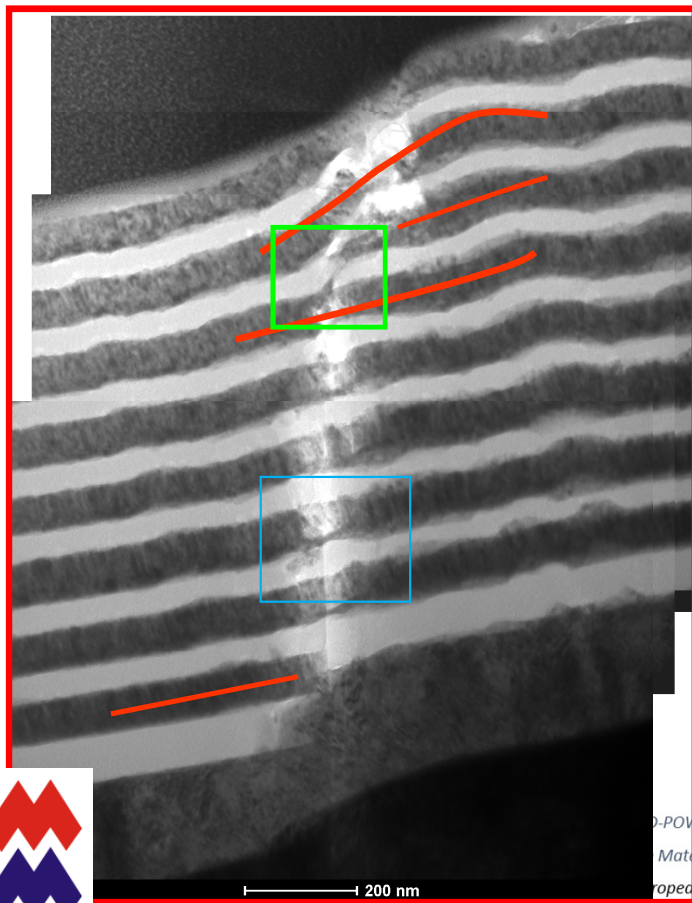
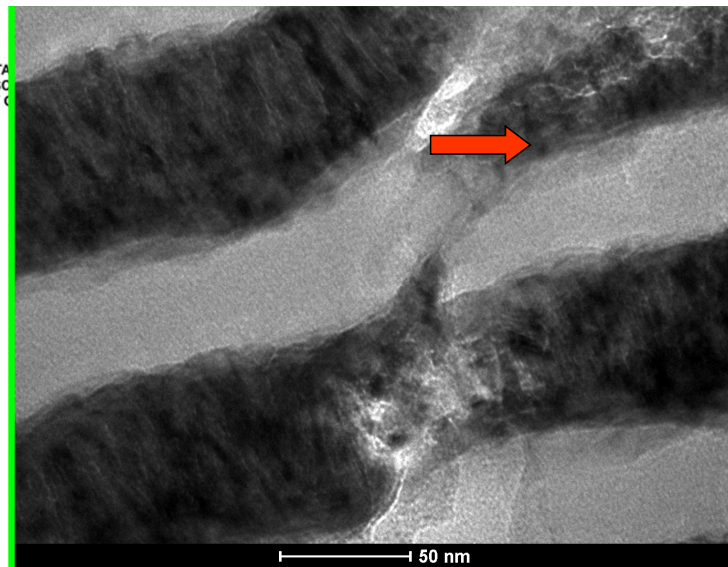


Eur
Fun
Know

TEM BF



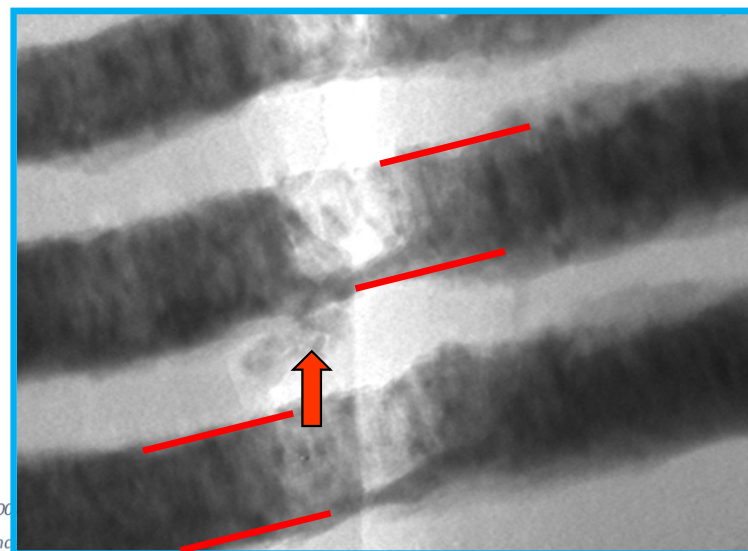
cracking

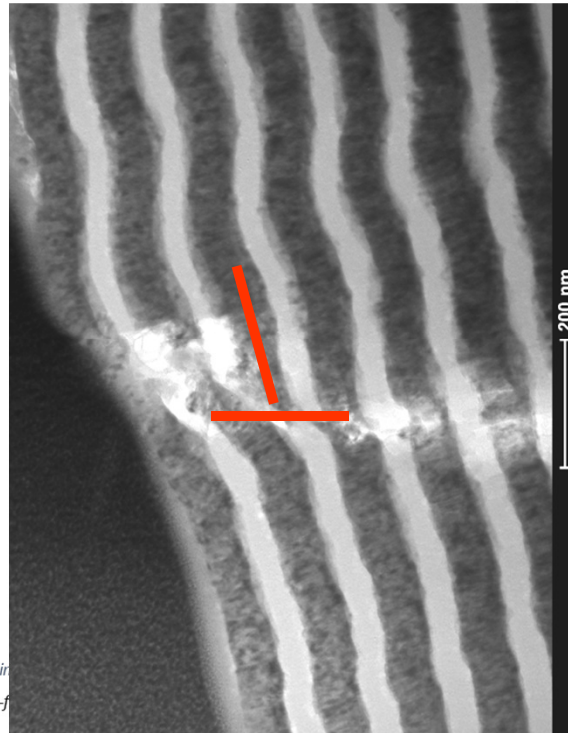
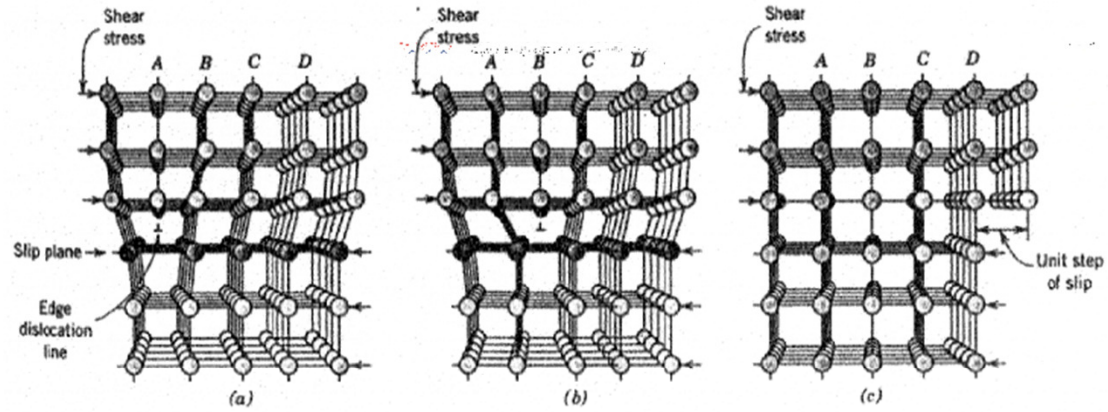


PO/R.03.02.00

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European Union within the European Social Funds



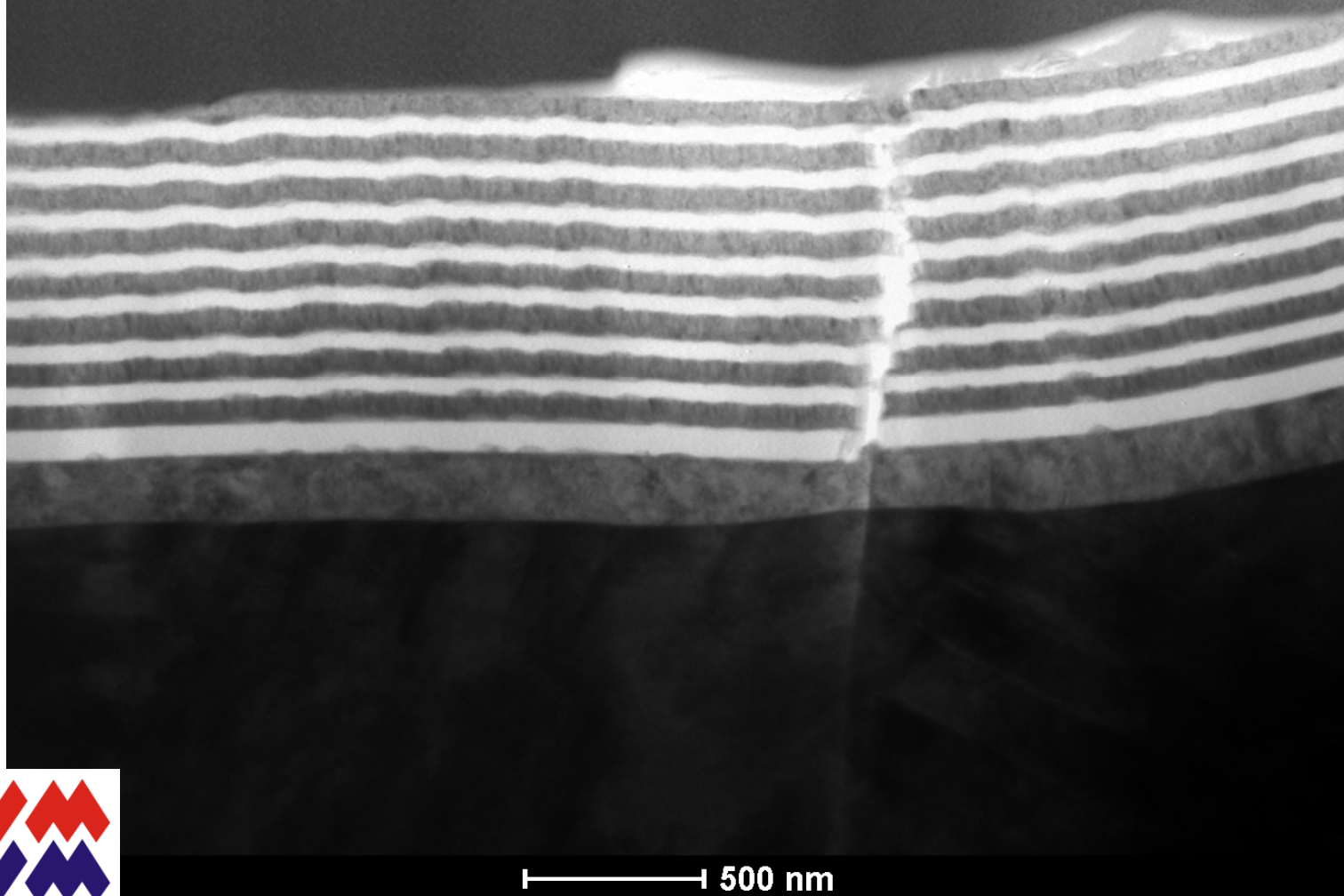




EU
FU
Kno

TEM *BF*

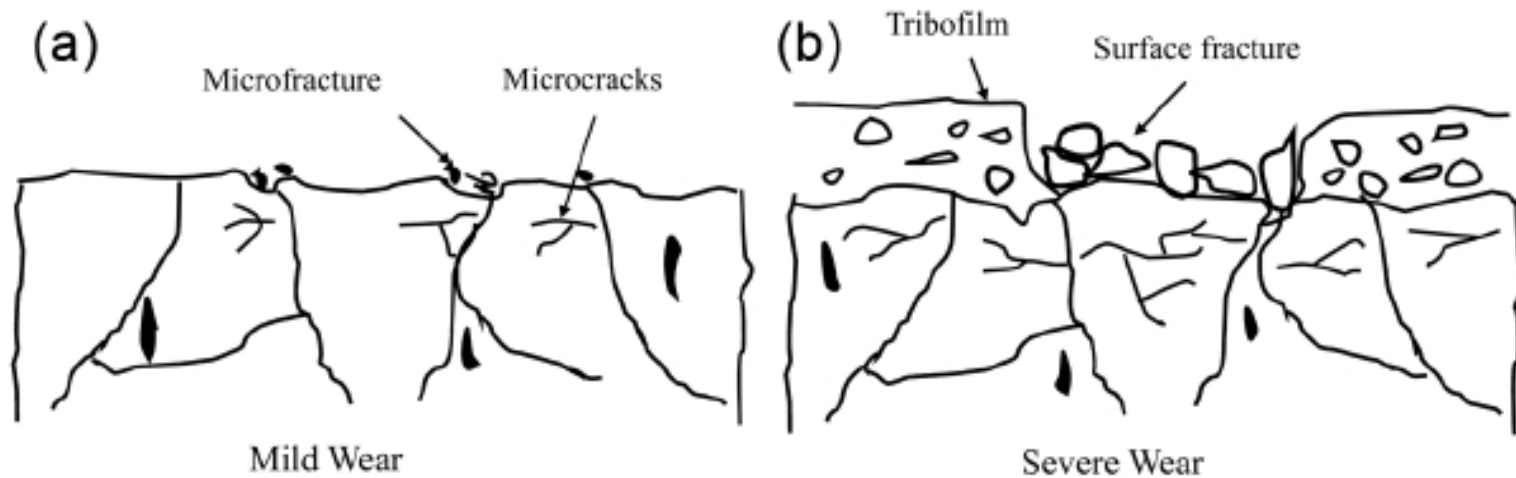
Layer by layer remove + tribo-film formation



500 nm



Microstructural characterization of the coatings by TEM after the wear test

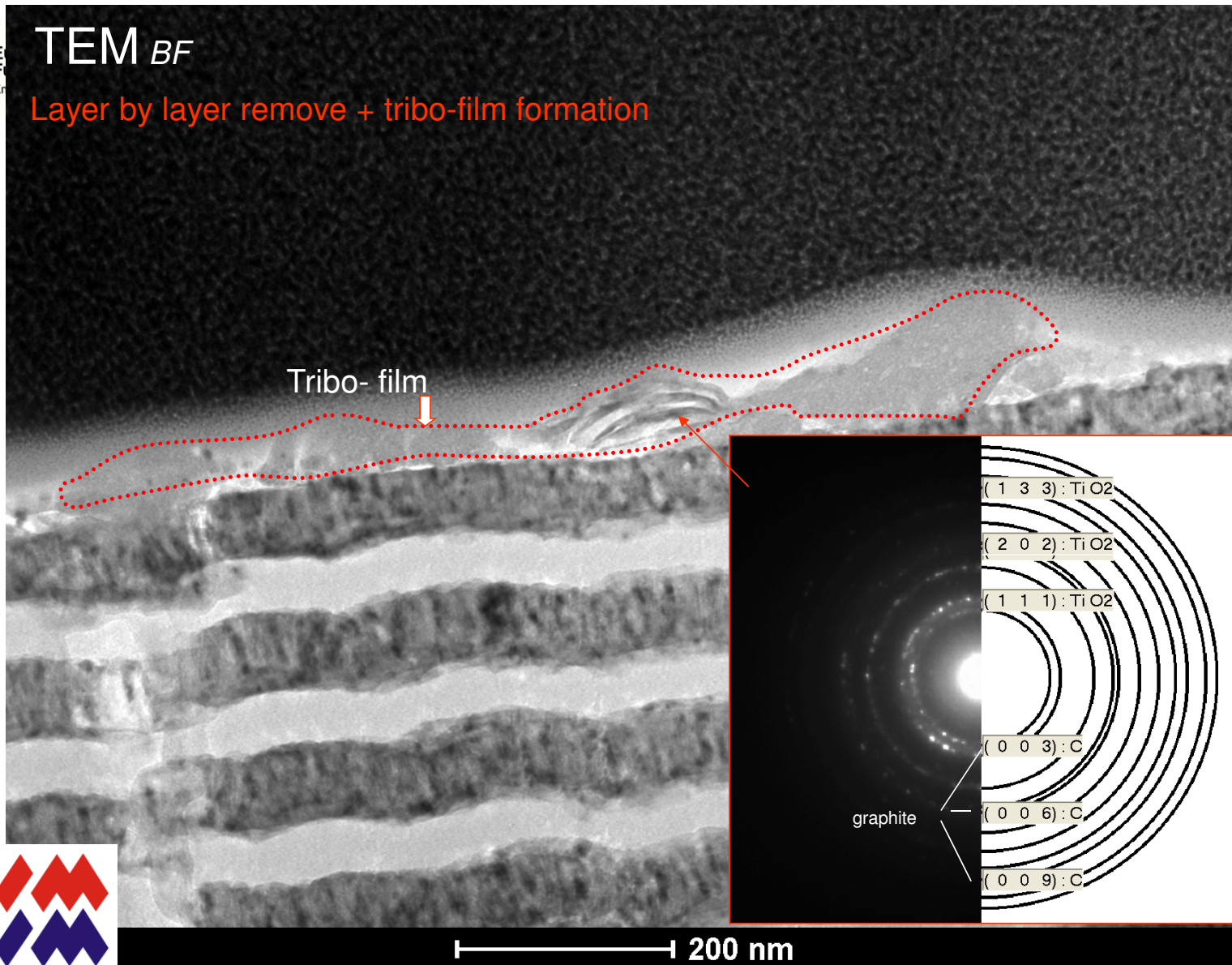




E
F
Kr

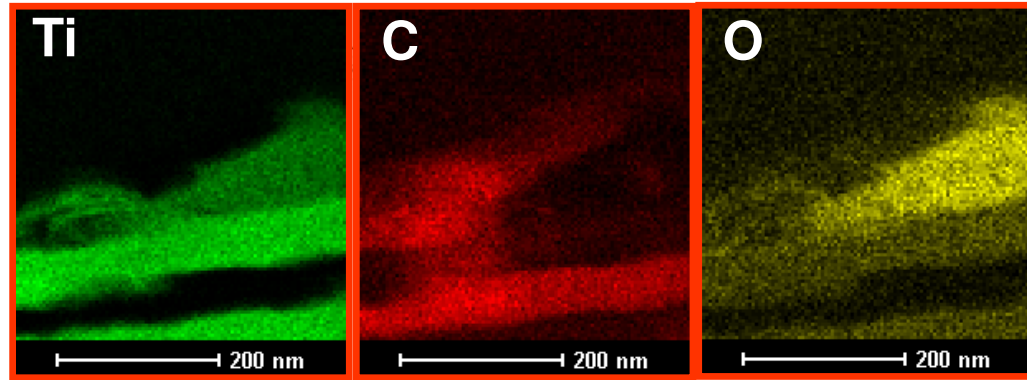
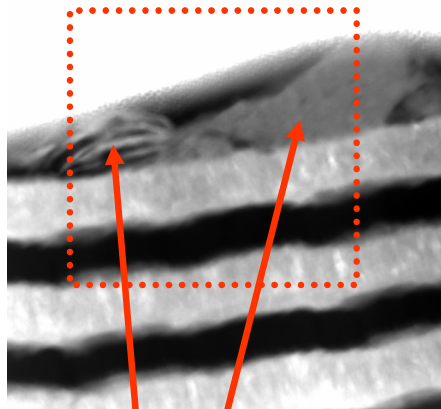
TEM_{BF}

Layer by layer remove + tribo-film formation

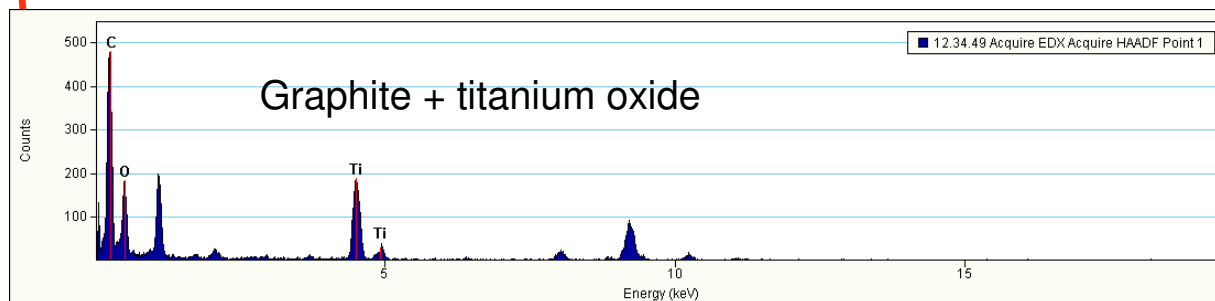
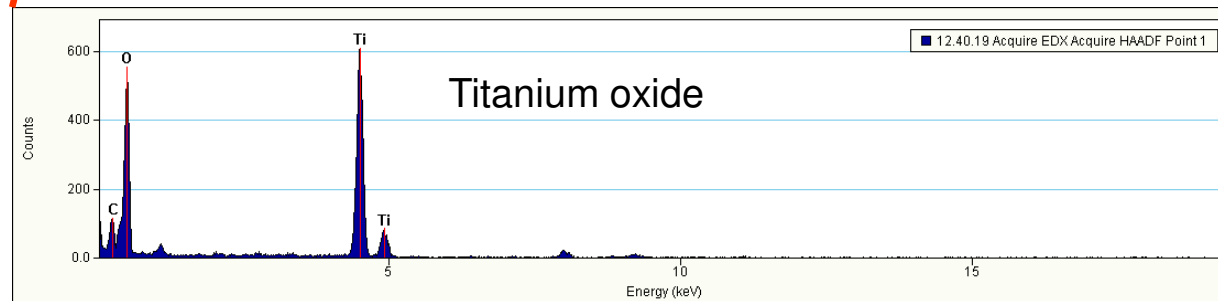


200 nm

EDS

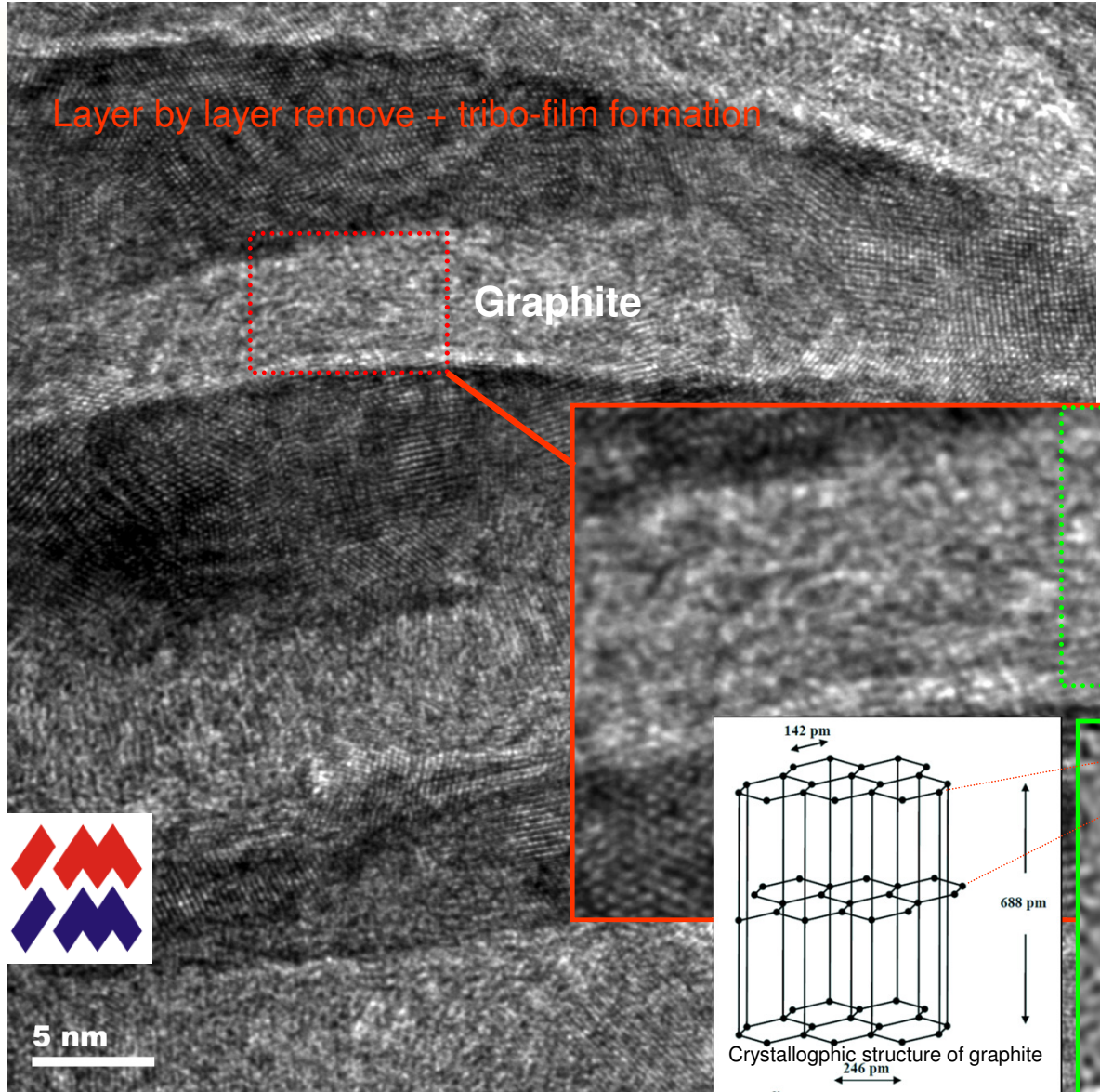


Layer by layer remove + tribo-film formation





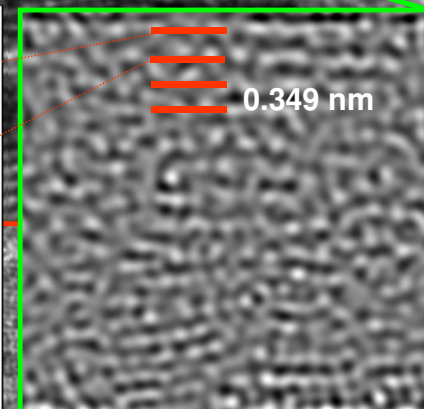
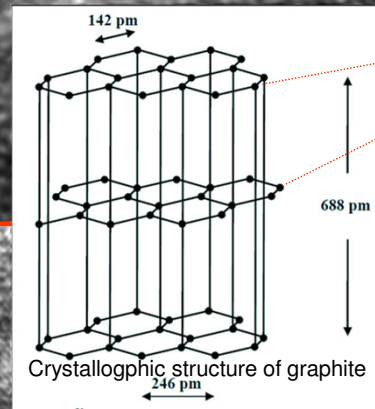
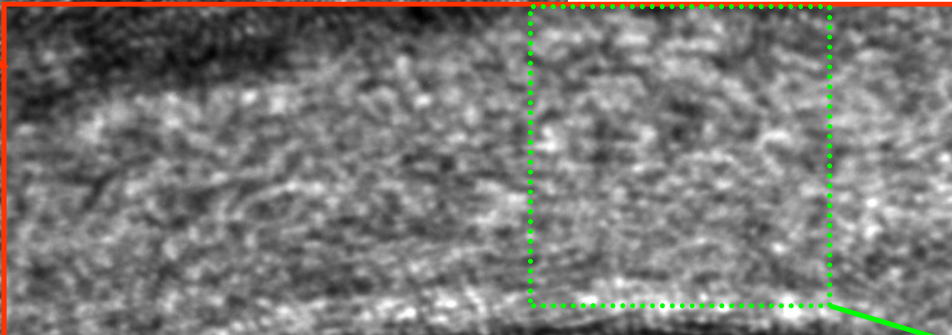
Layer by layer remove + tribo-film formation



HRTEM
Graphite
+ titanium oxide



5 nm



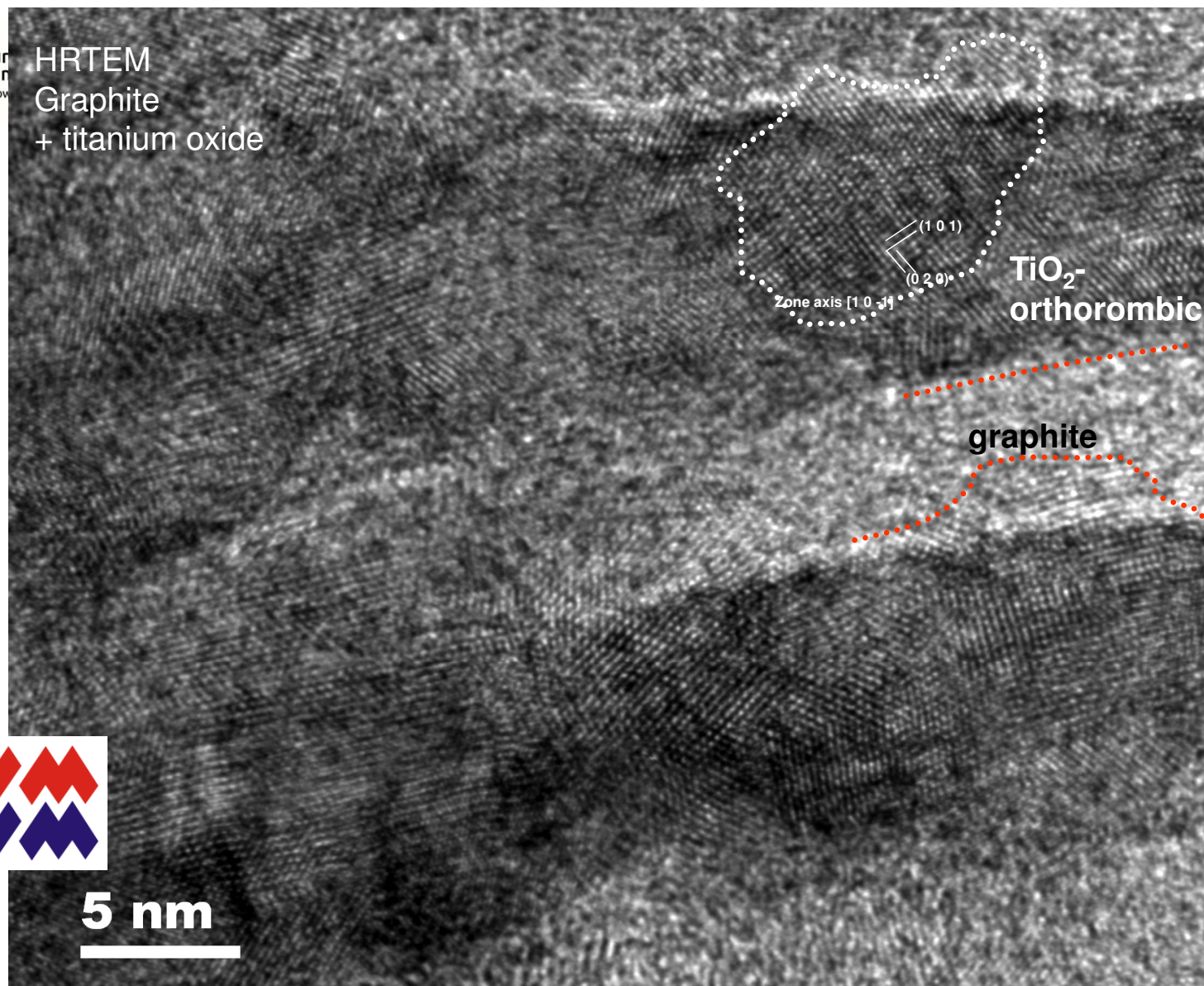


Eur
Fun
Know

HRTEM
Graphite
+ titanium oxide



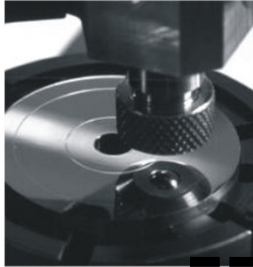
5 nm



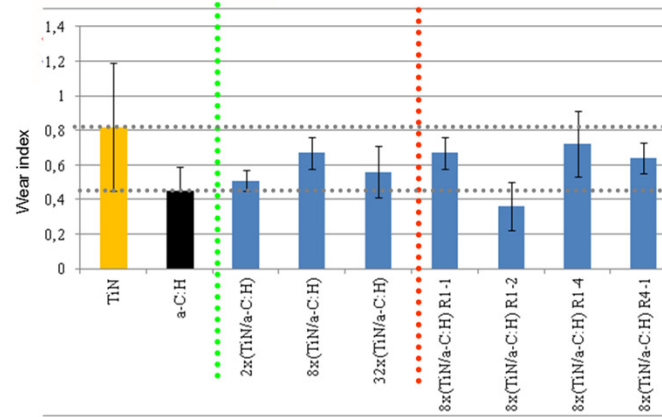


European Funds
Knowledge Education Development

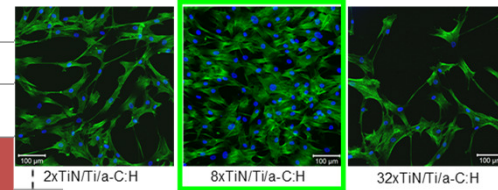
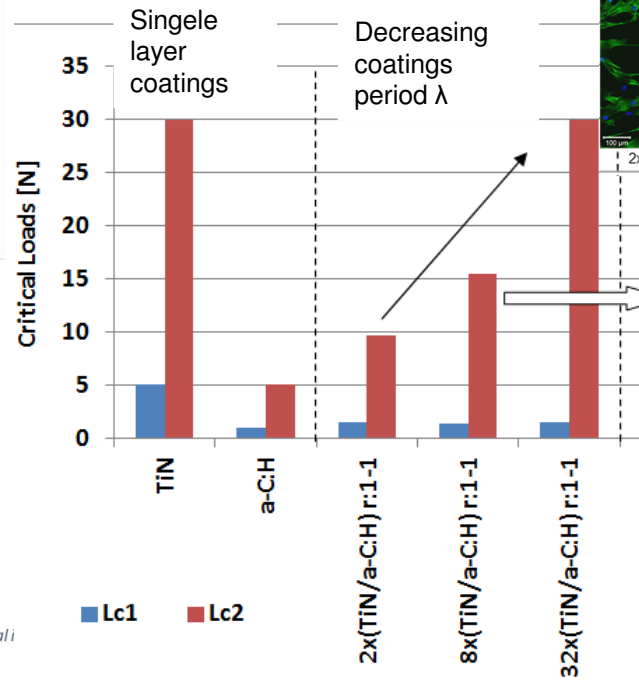
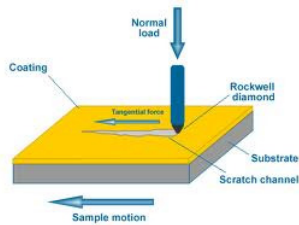
Ball-on-disc Test



European Union
European Social Fund



SCRATCH TEST

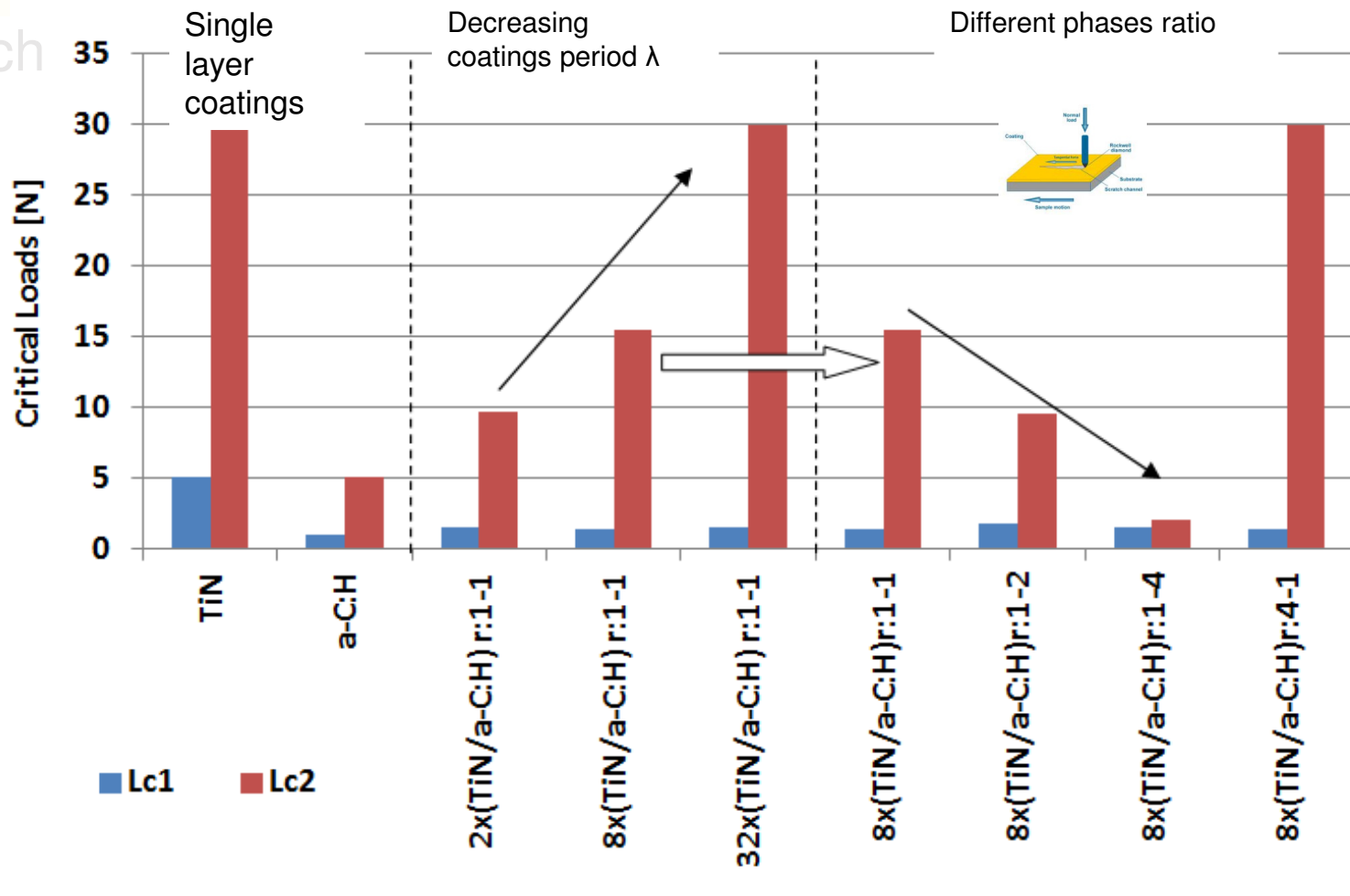


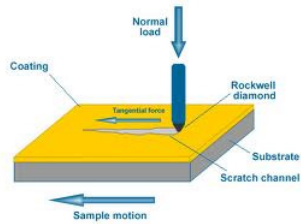
International i

tion



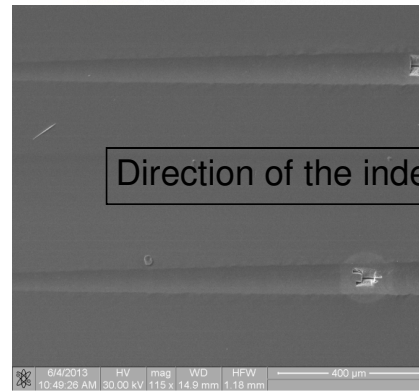
Scratch
test



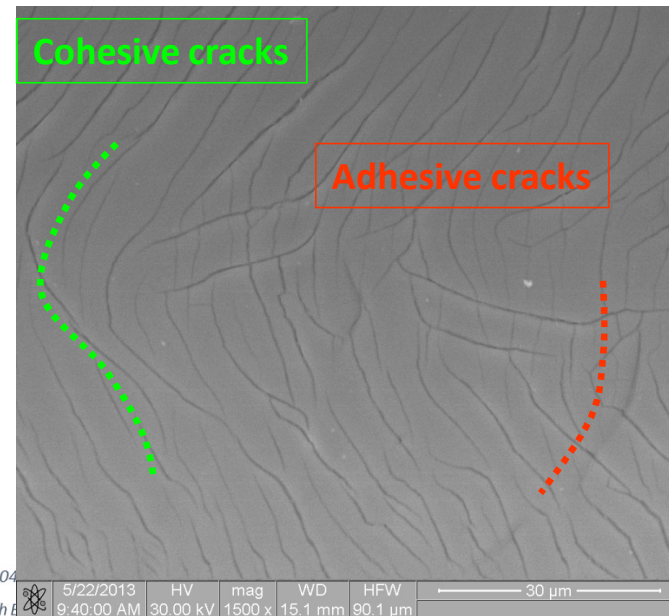
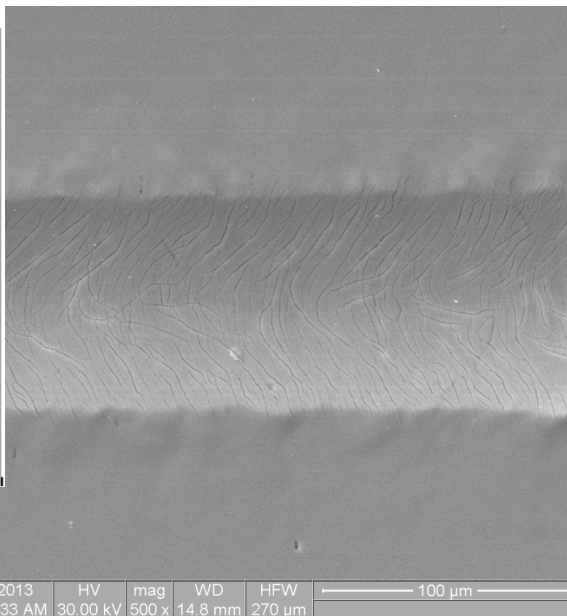
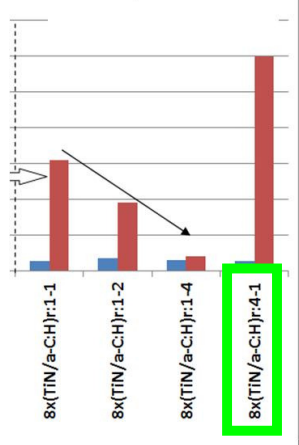


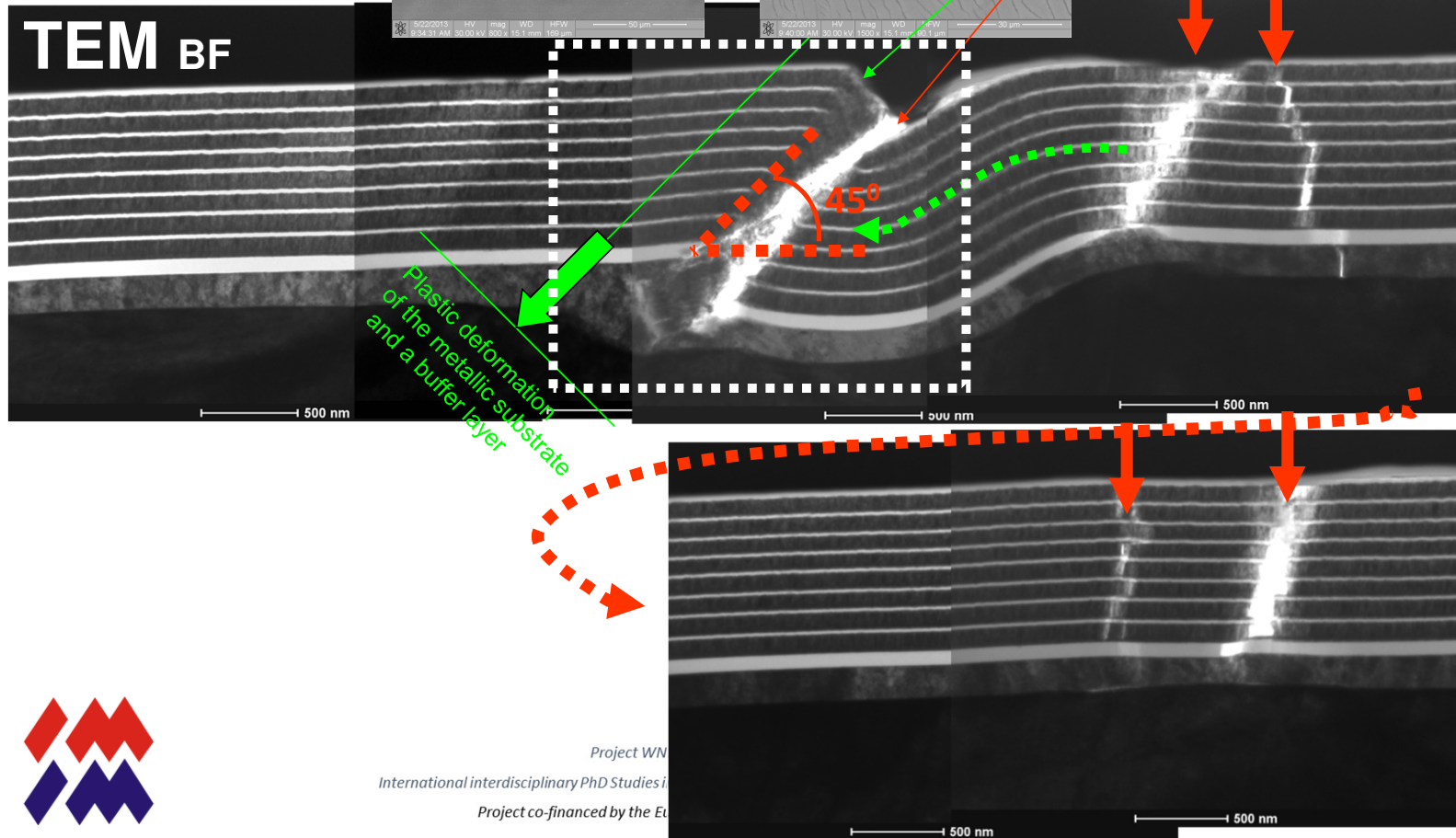
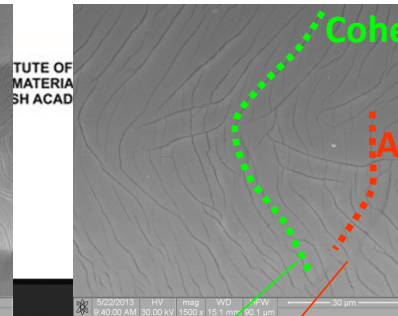
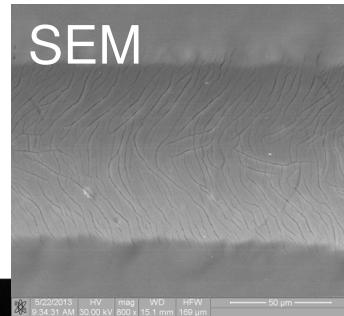
SEM

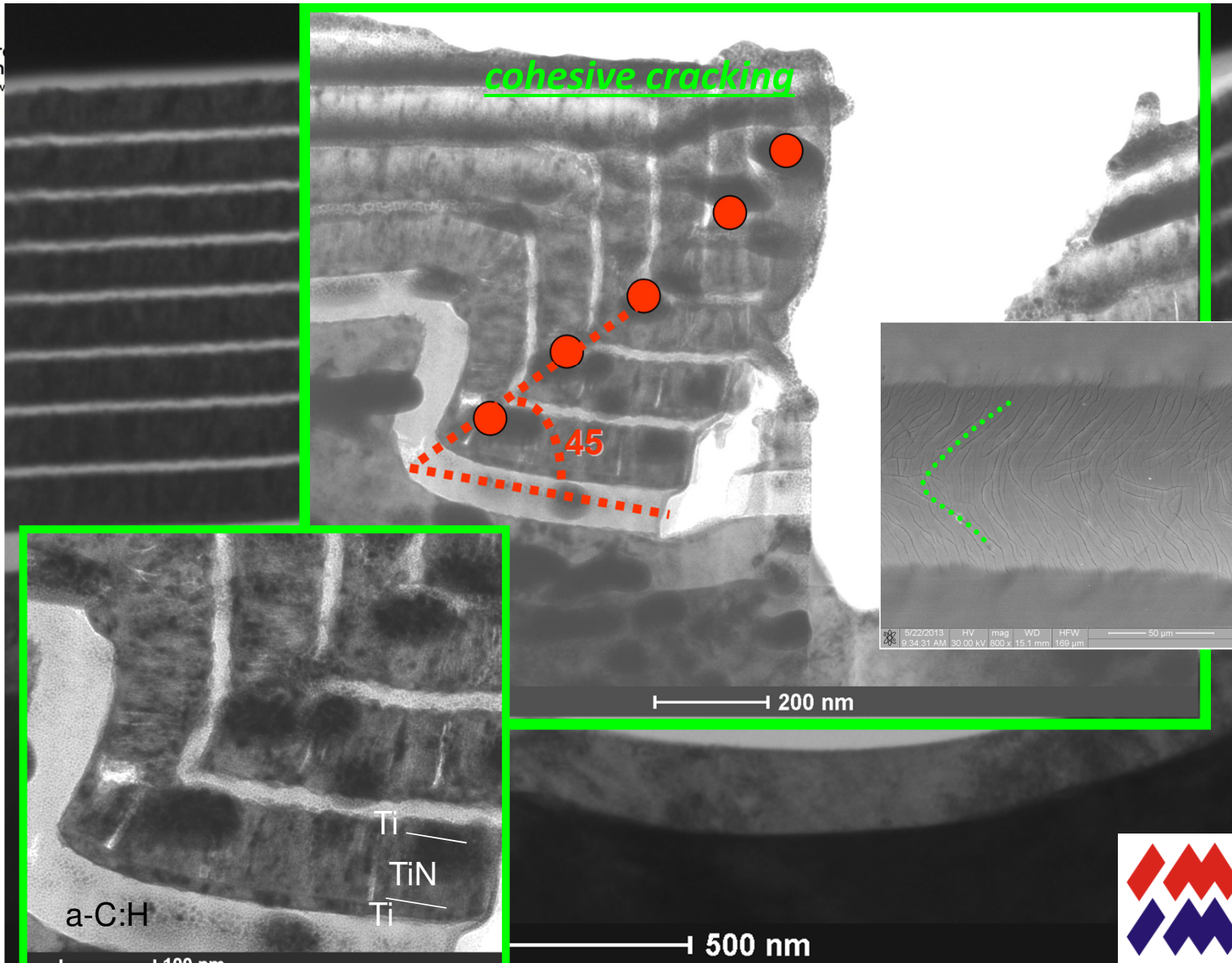
Direction of the indenter movement

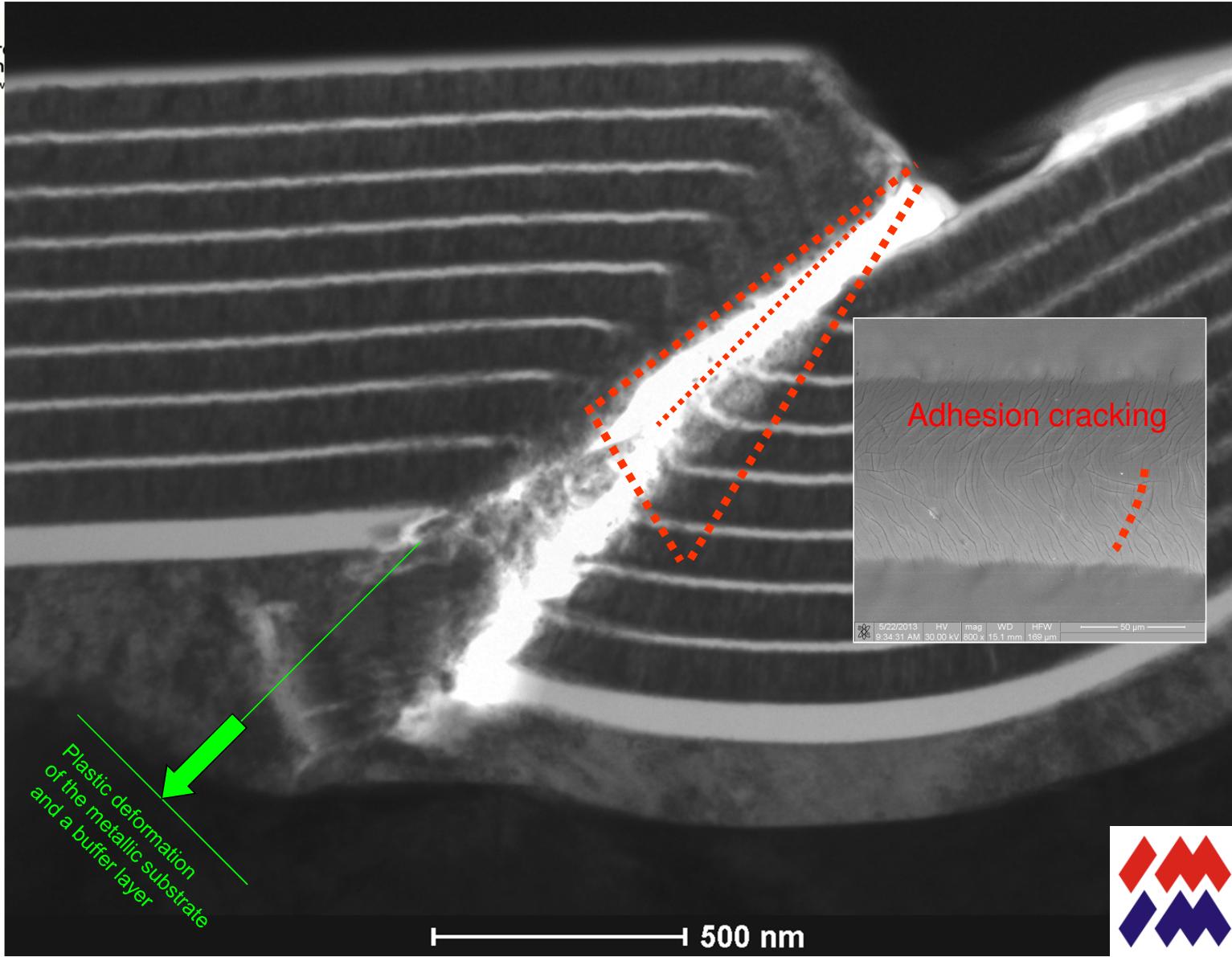


Different phases ratio





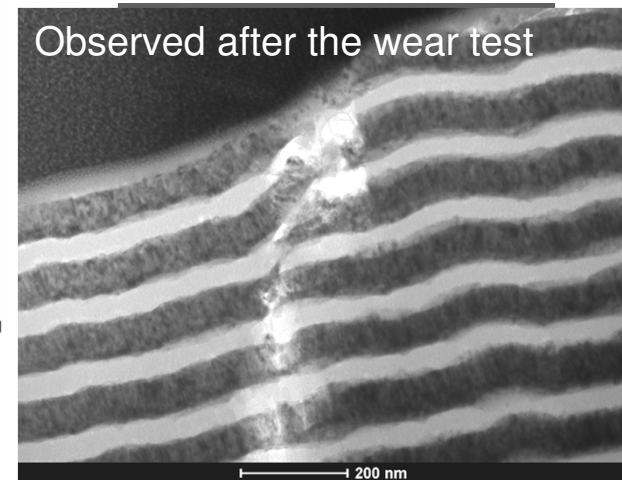
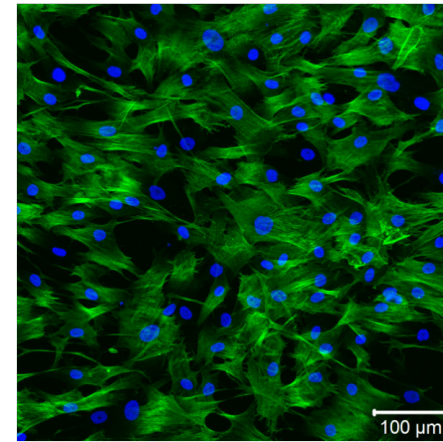
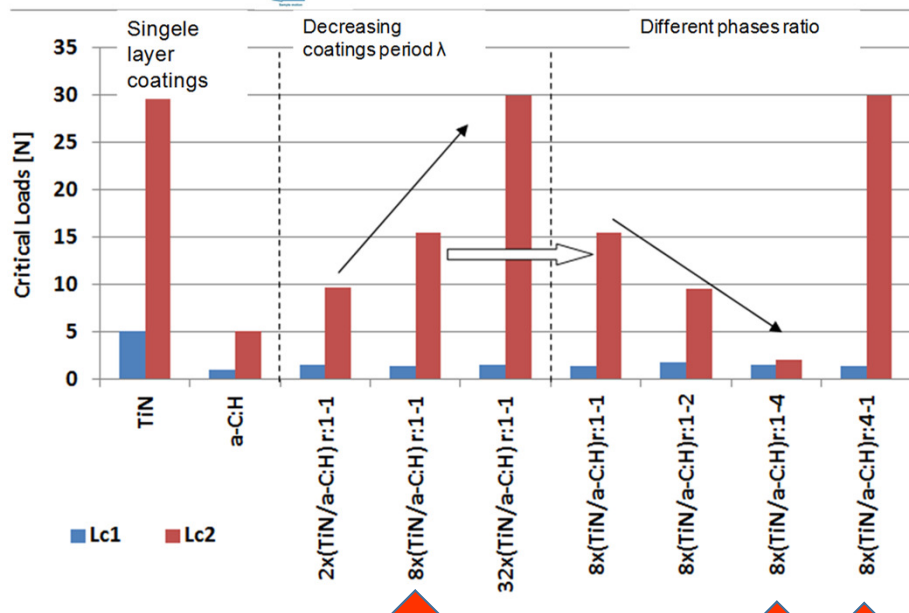






Summary part: I

Scratch test



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- Project NCN nr: 3066/B/T02/2011/40- *FINISHED*
- Project NCN nr: 2012/06/M/ST8/00408- *HARMONIA- FINISHED*
- Project NCN nr: 2012/07/B/ST8/03396- *OPUS- FINISHED*
- Project NCN nr: 2014/15/B/ST8/00103- *OPUS- FINISHED*
- Project NCN nr: 2015/19/B/ST8/00942- *OPUS- in progress*
- Project NCBR, number: DZP/M-ERA.NET-2015/285/2016- *in progress*

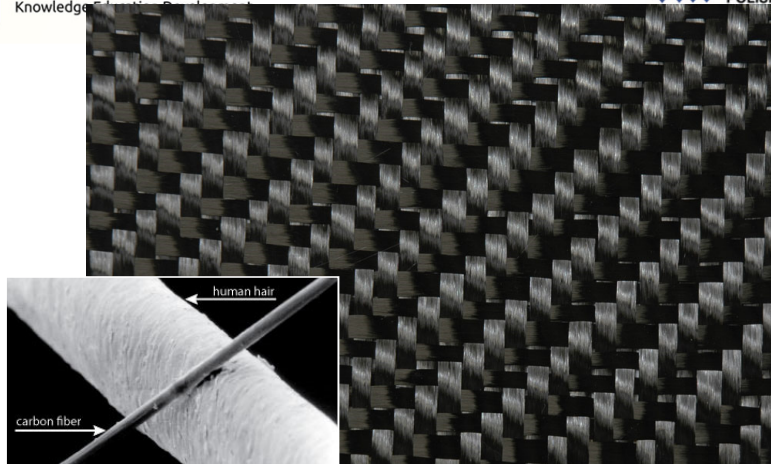


Title: Development of wear resistant multilayer protective coatings for carbon/ fiber composites

Project WND-POWR.03.02.00-00-1043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

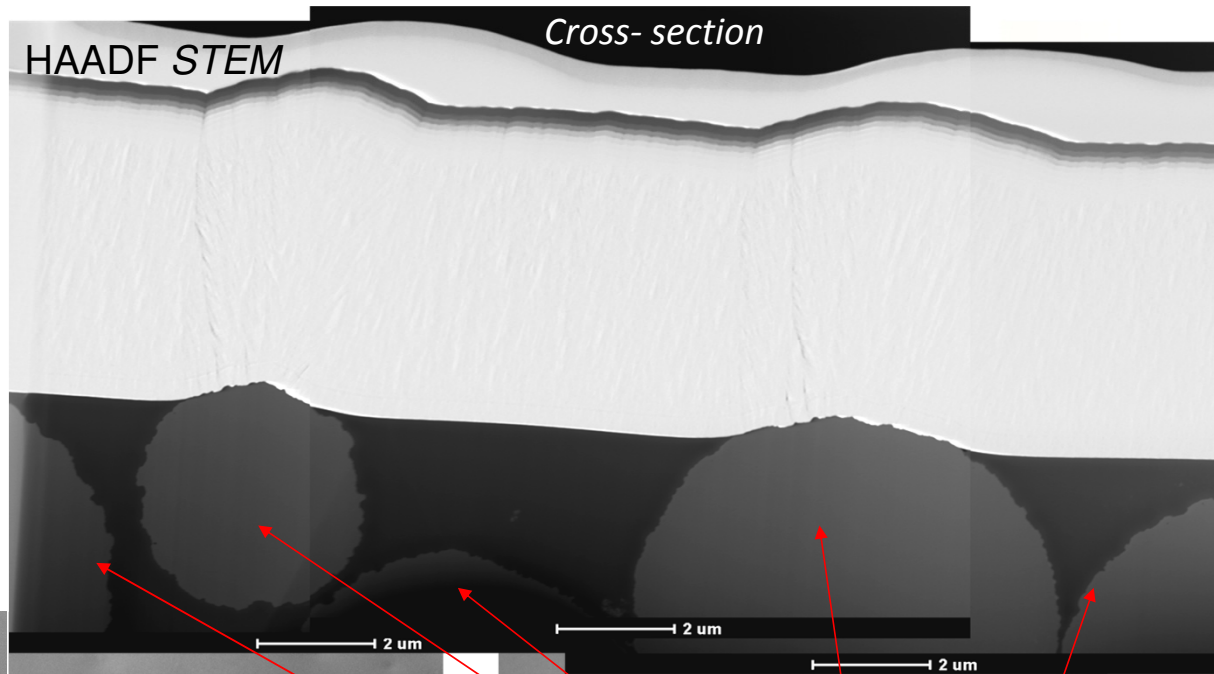
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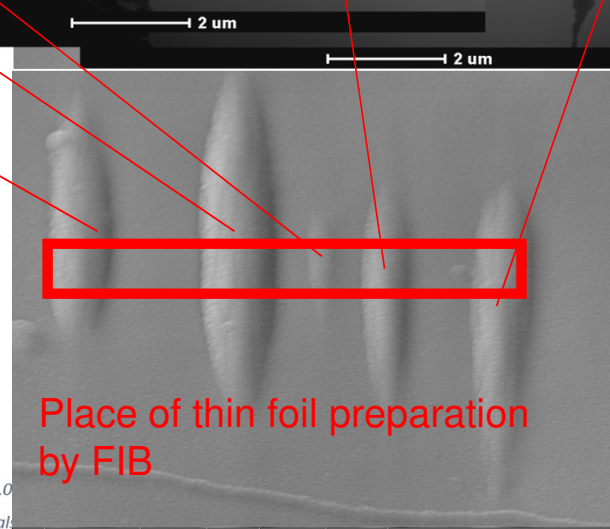
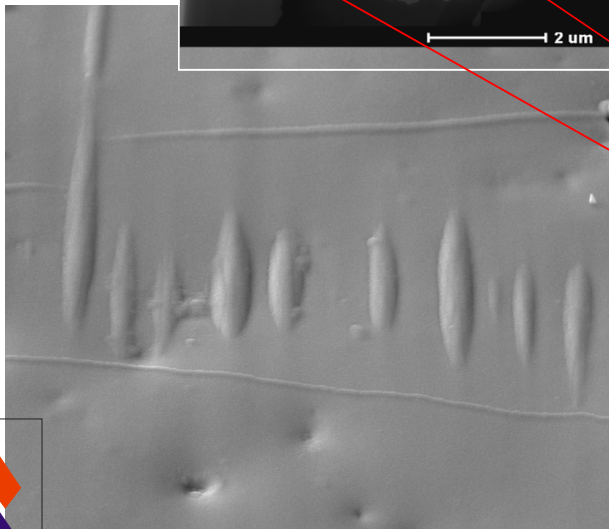
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International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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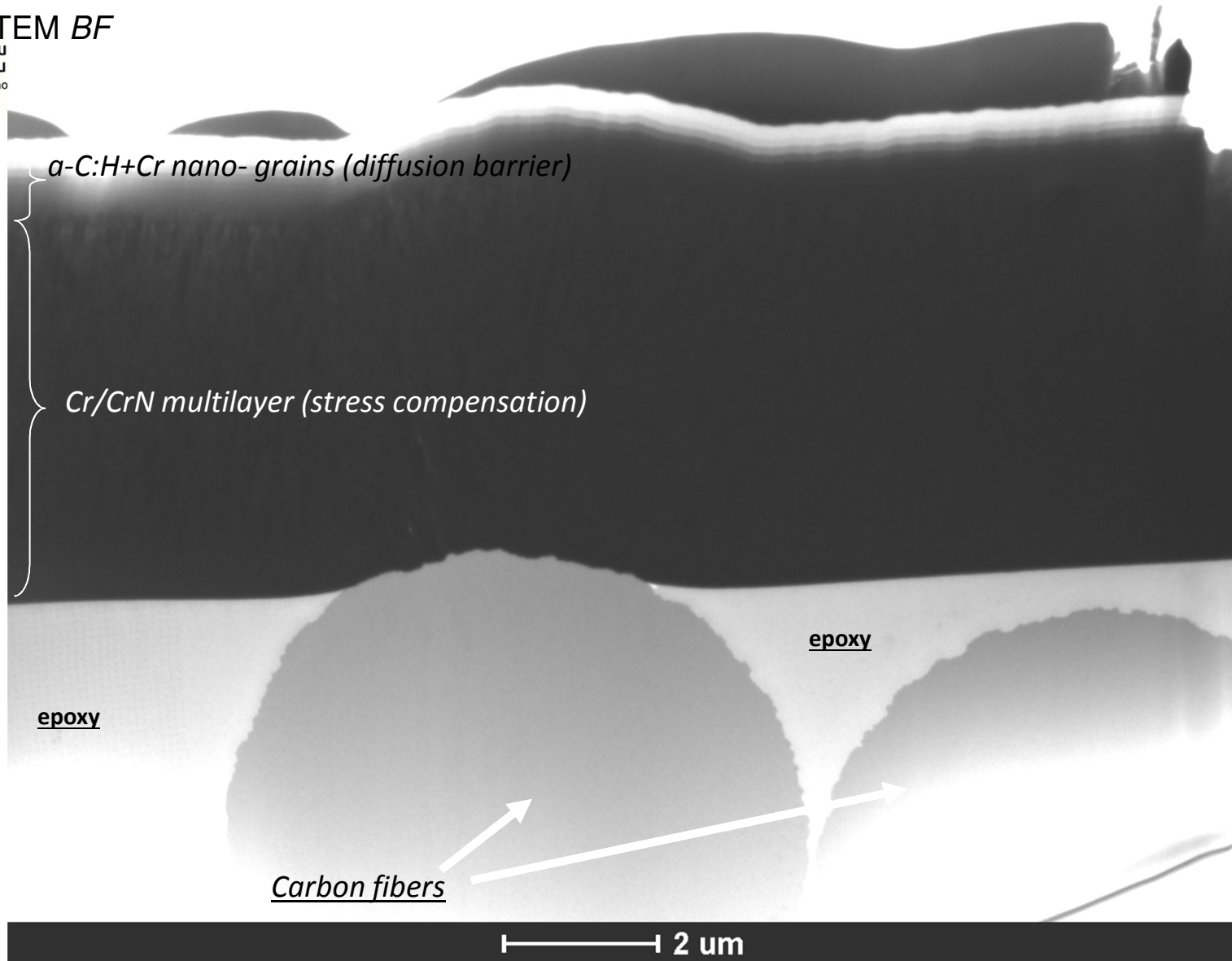


SEM



Place of thin foil preparation
by FIB

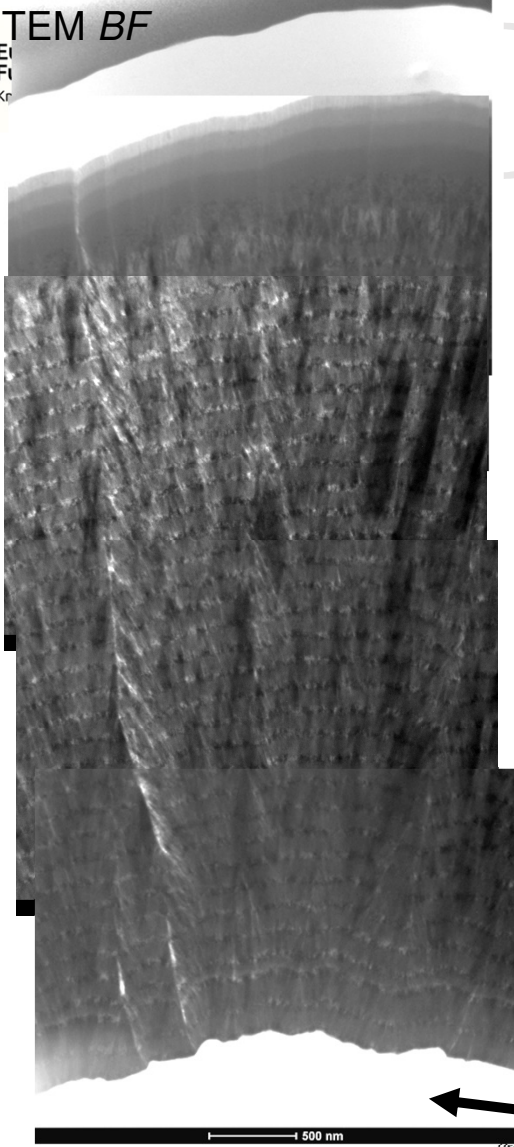






TEM BF

ET
FI
Kr



a-C:H+
Cr
nano particles

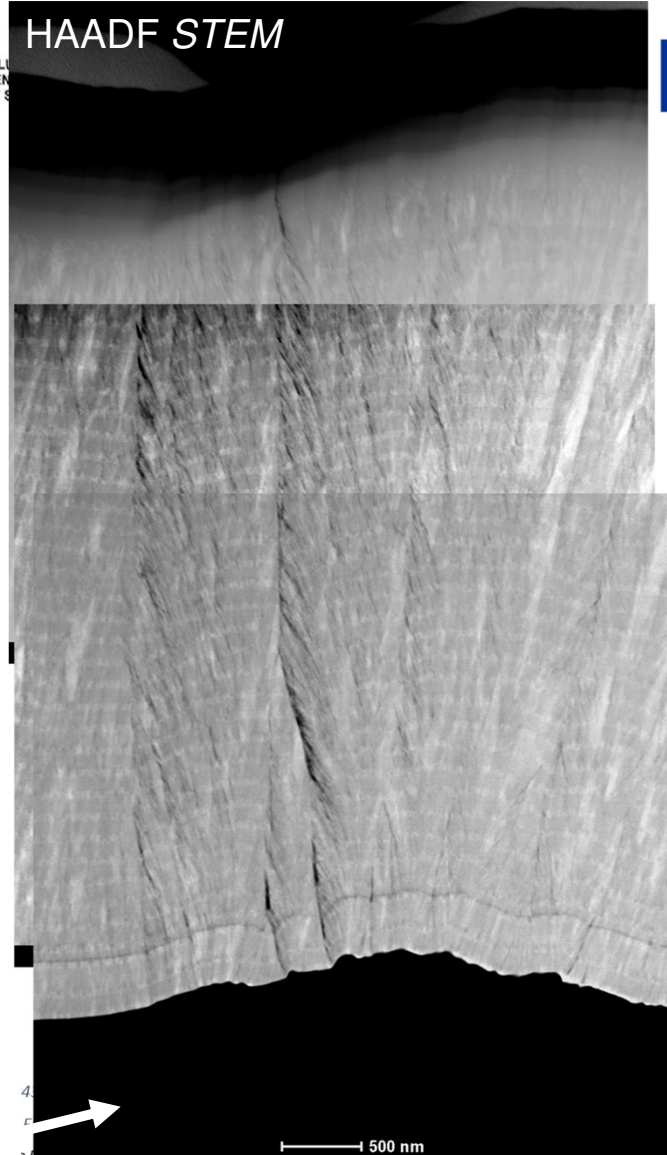
Cr/CrN
multilayer

Carbon Fiber

500 nm

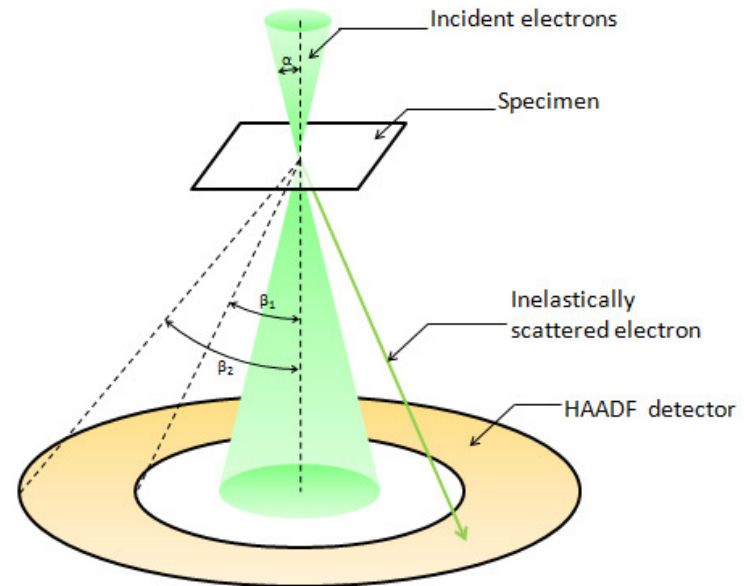
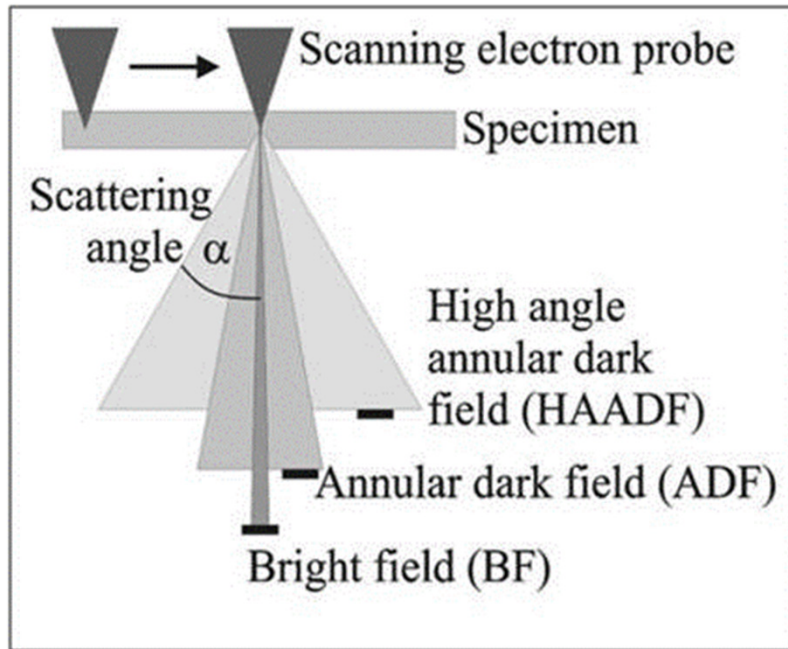
ETALL
SCIEN
TY OF S

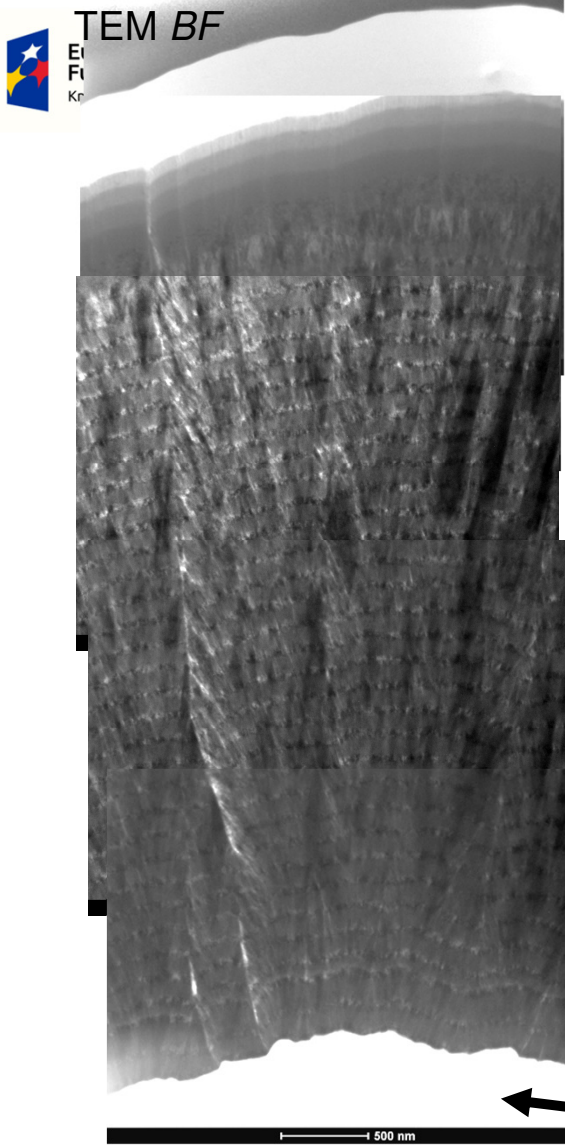
HAADF STEM



500 nm

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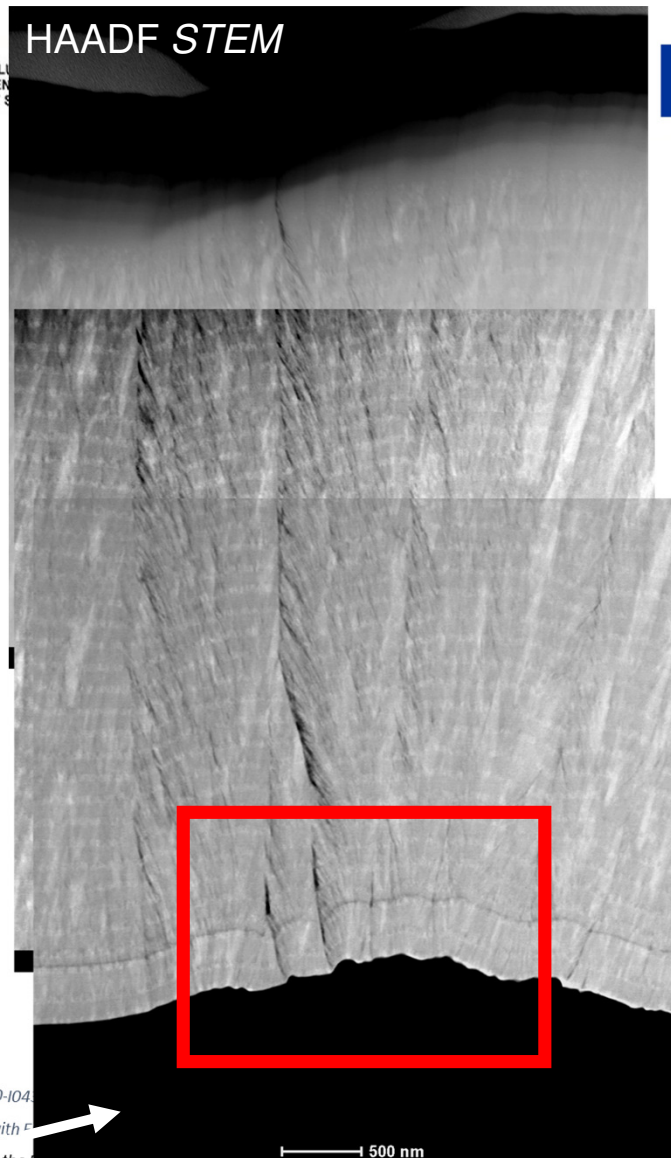
TEM BF



a-C:H+
Cr
nano particles

Cr/CrN
multilayer

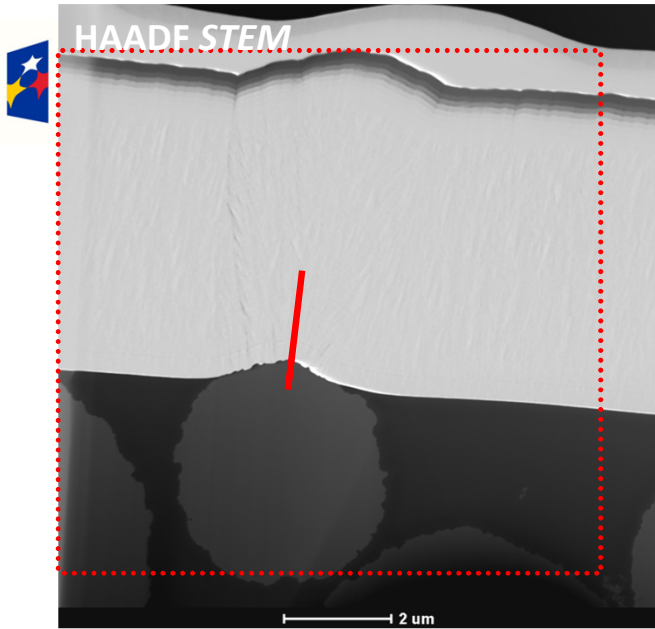
Carbon Fiber



HAADF STEM

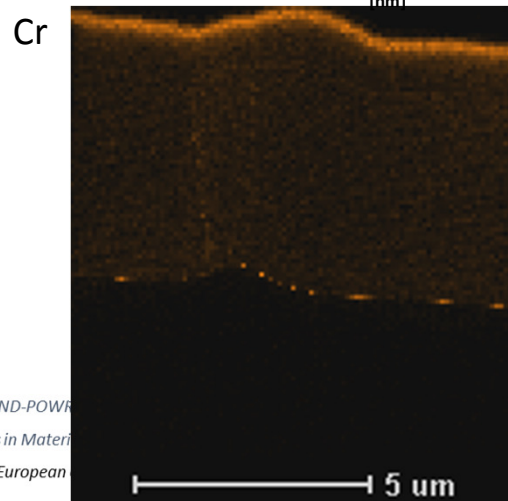
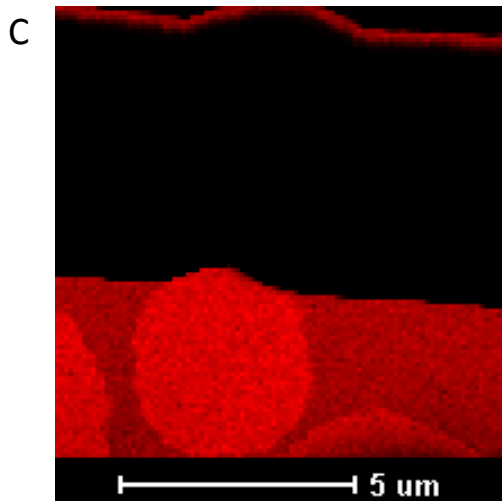
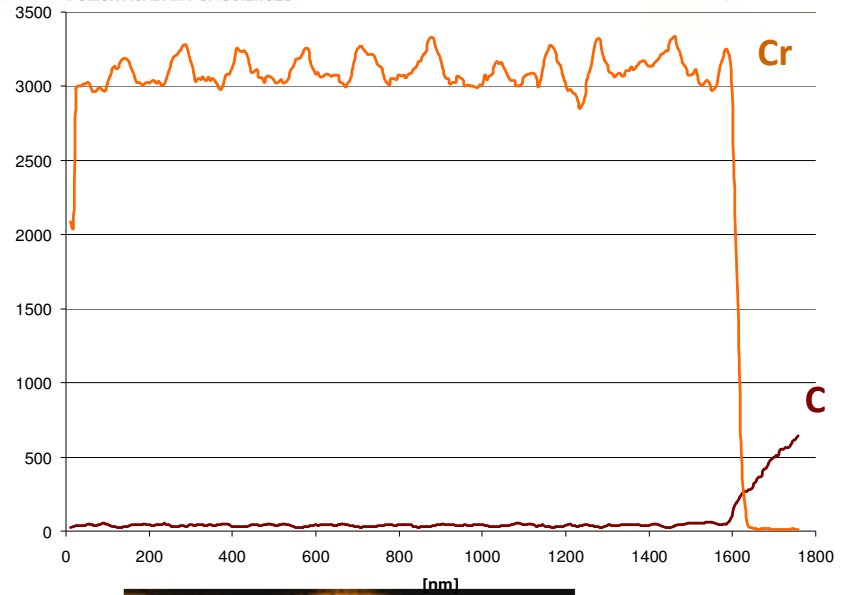


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INSTITUTE OF METALLURGY
AND MATERIALS SCIENCE
POLISH ACADEMY OF SCIENCES

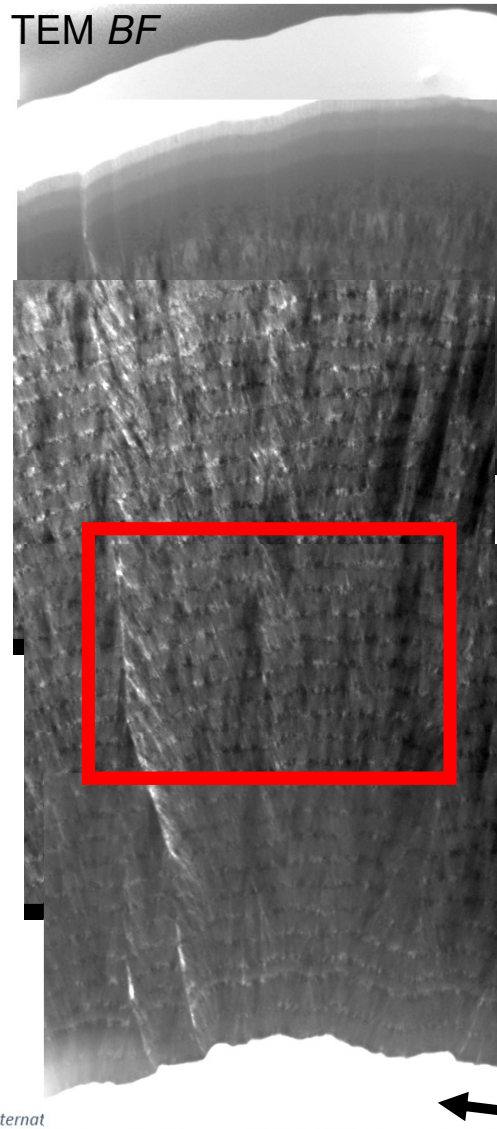
European Union
European Social Fund



EDS

Project WND-POWER
PhD Studies in Materials
Supported by the European





CES

a-C:H+
Cr
nano particles

Cr/CrN
multilayer

Carbon Fiber

Internat

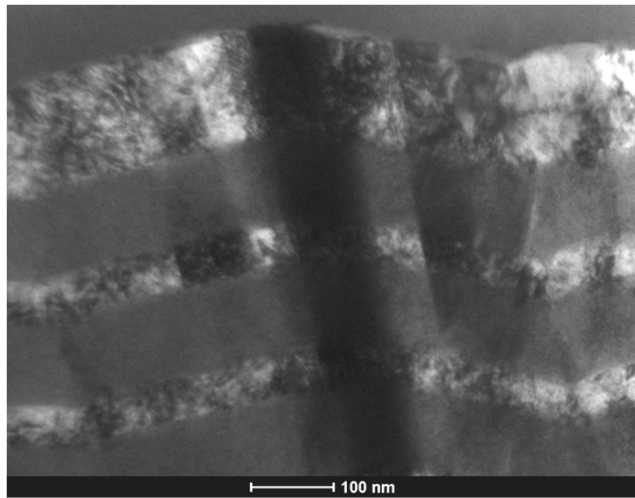
500 nm

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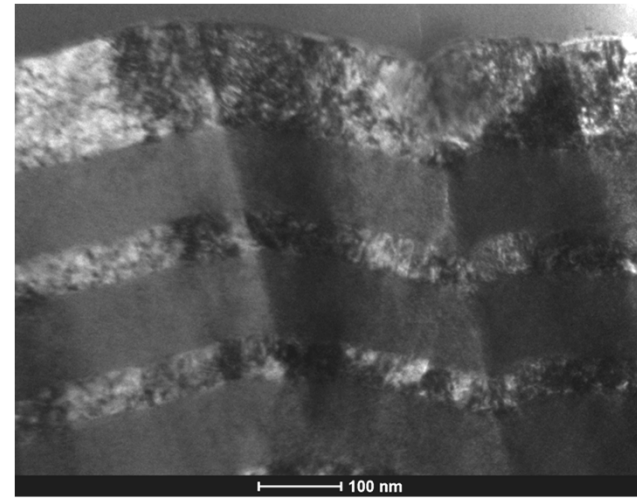




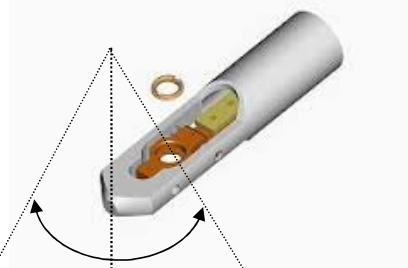
TEM BF



$\alpha = 0^\circ$



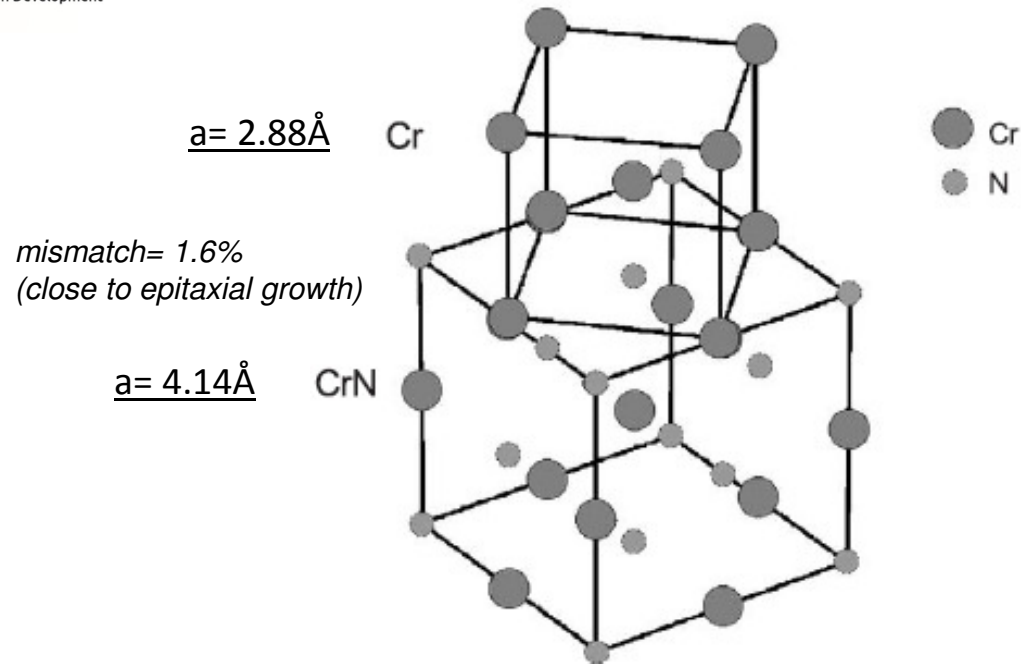
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- Project WNI 2019-03-02-00-1043/16 +

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Surface & Coatings Technology 188–189 (2004) 338–343

**SURFACE
& COATINGS
TECHNOLOGY**

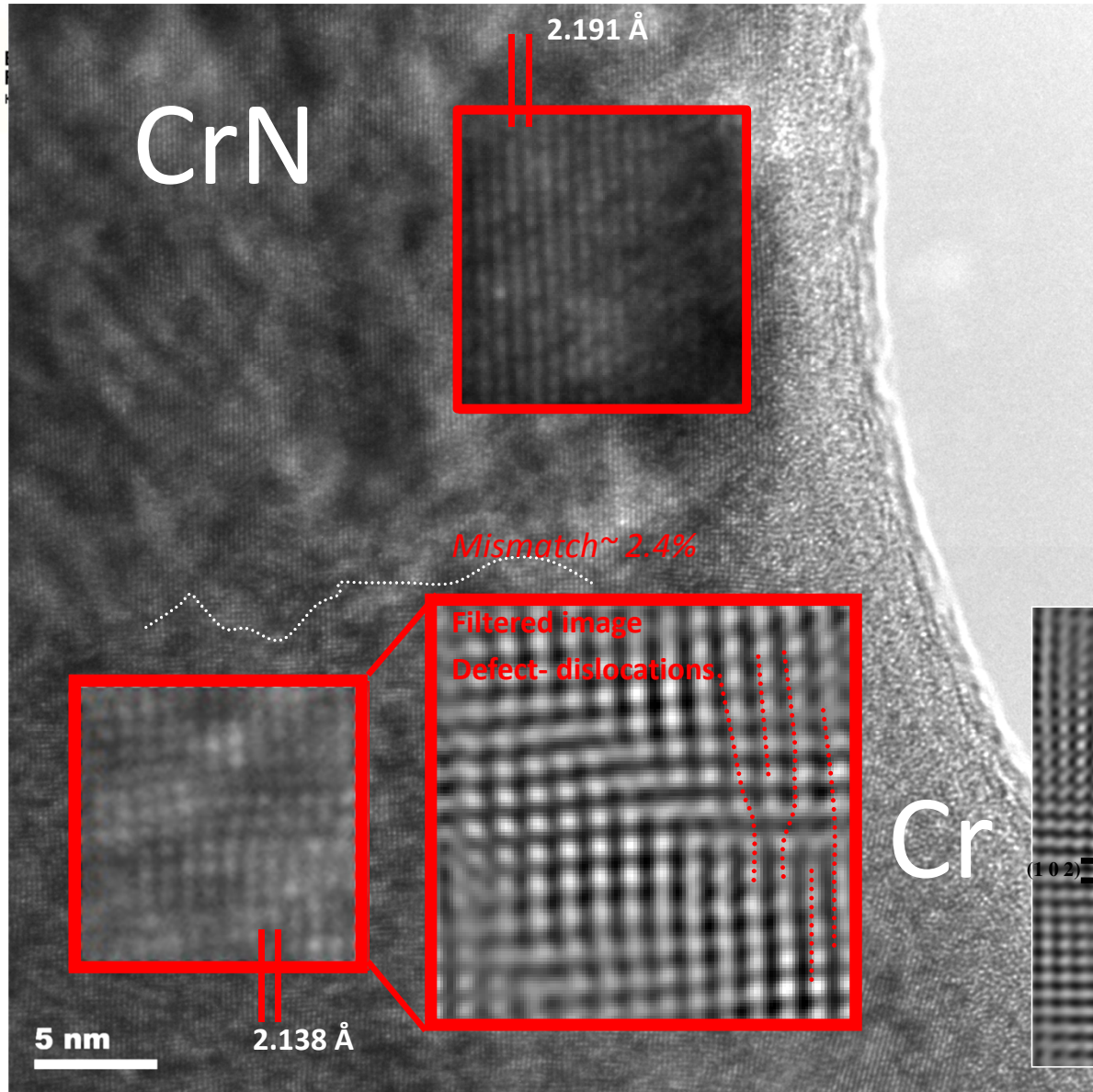
www.elsevier.com/locate/surfcoat

Period dependence of hardness and microstructure on nanometric Cr/CrN multilayers

J. Romero*, J. Esteve, A. Lousa

Departamento Física Aplicada i Òptica, Universitat de Barcelona, Avda. Diagonal 647, E-08028 Barcelona, Catalunya, Spain

Available online 12 October 2004



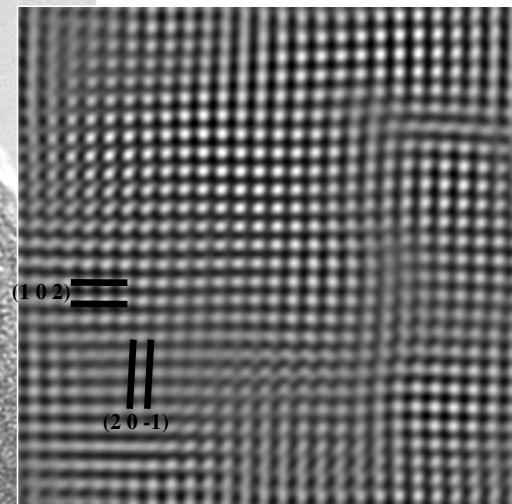
European Union
European Social Fund

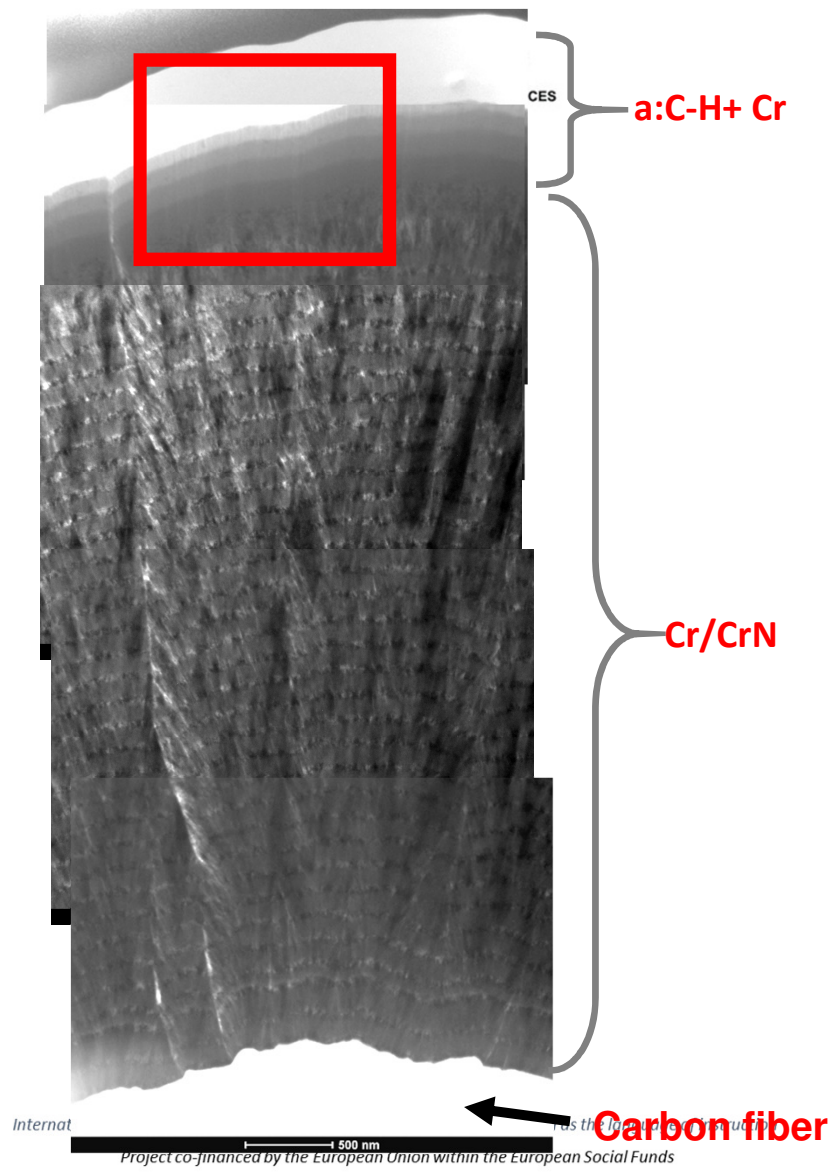


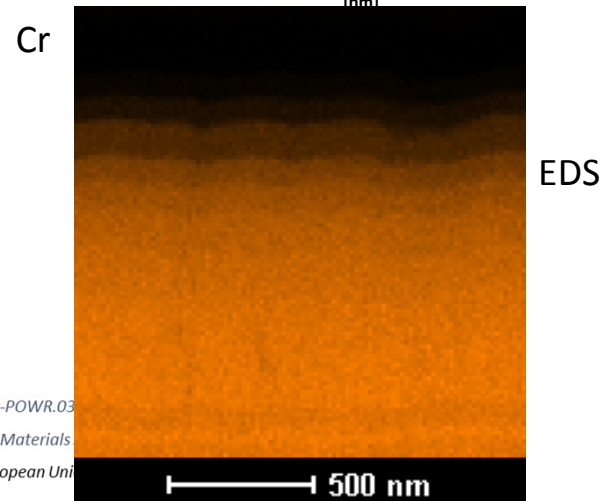
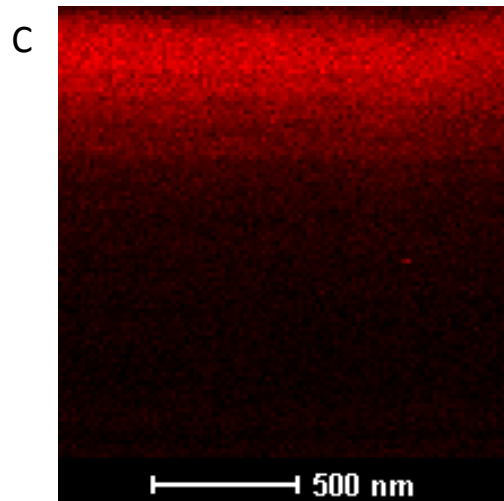
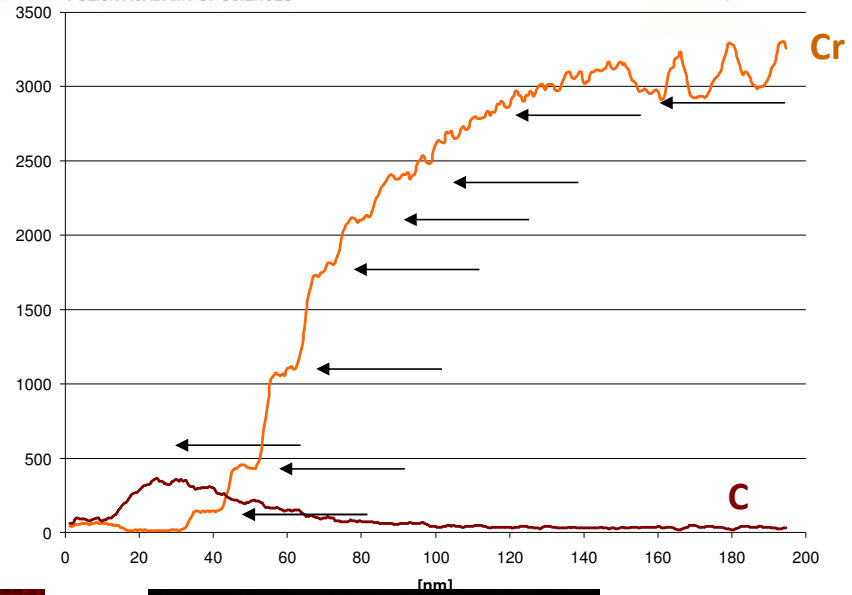
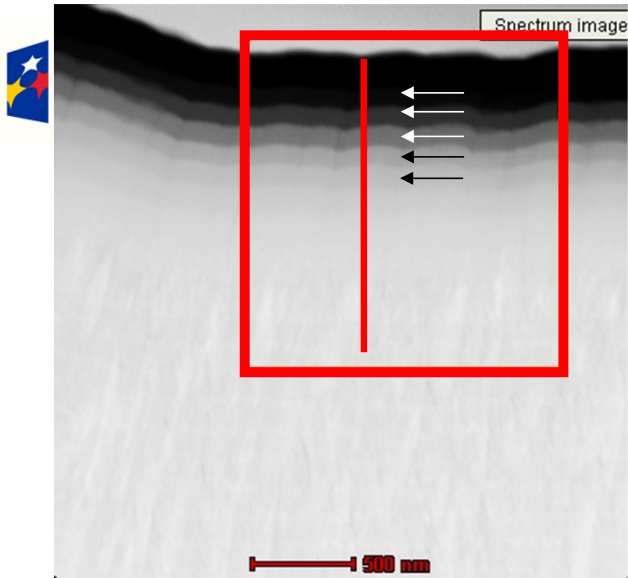
HRTEM

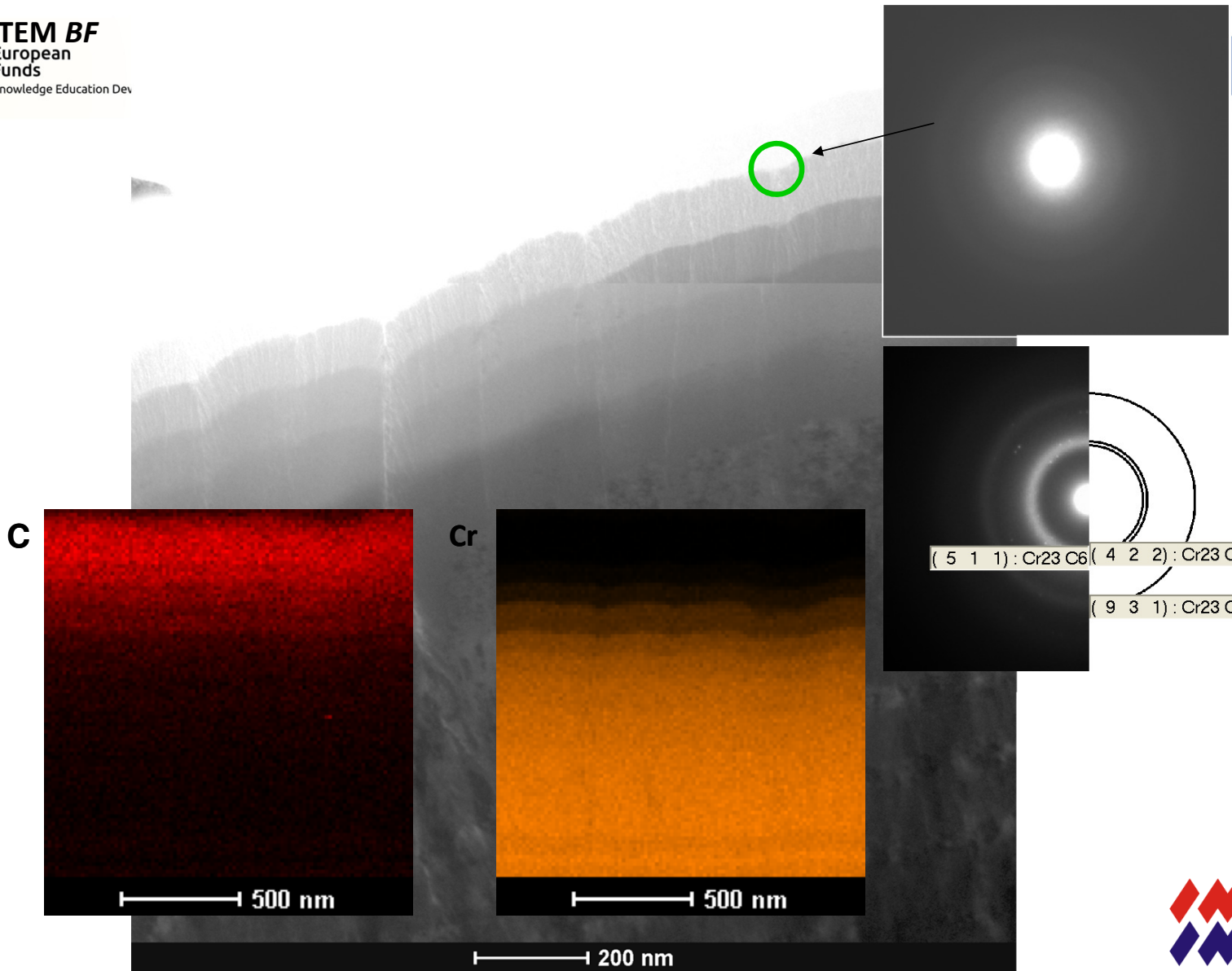


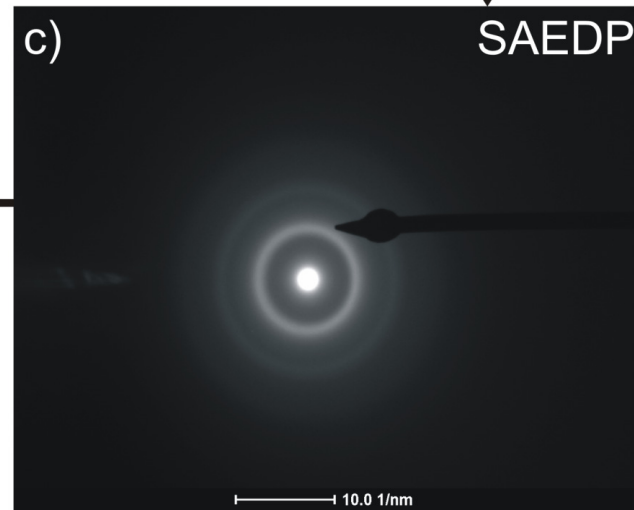
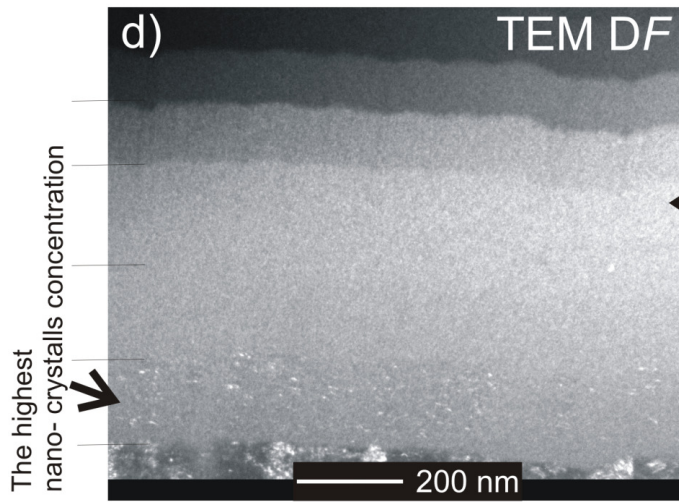
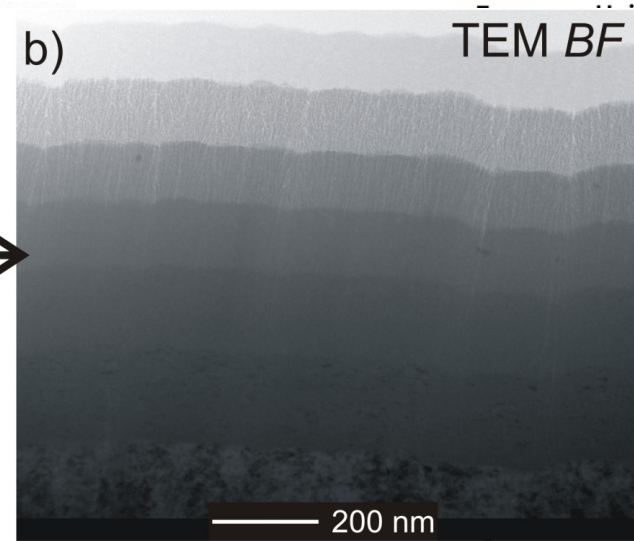
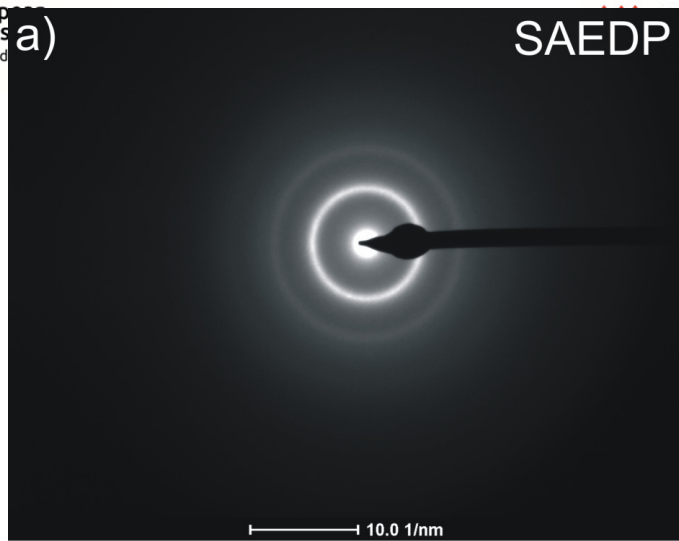
Zone axis [0 1 0]
Cr cubic

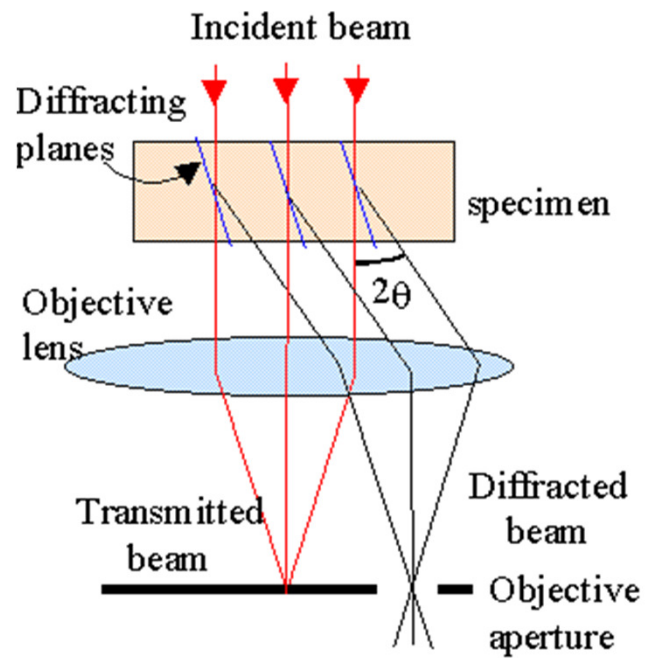




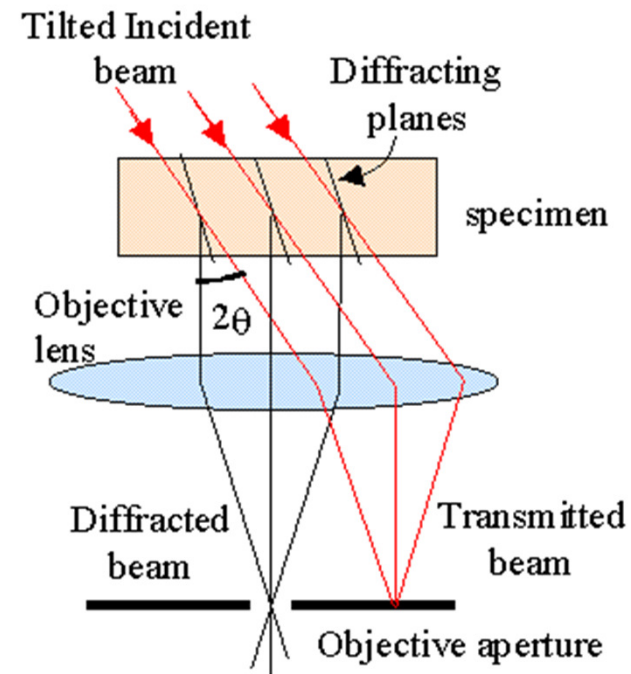




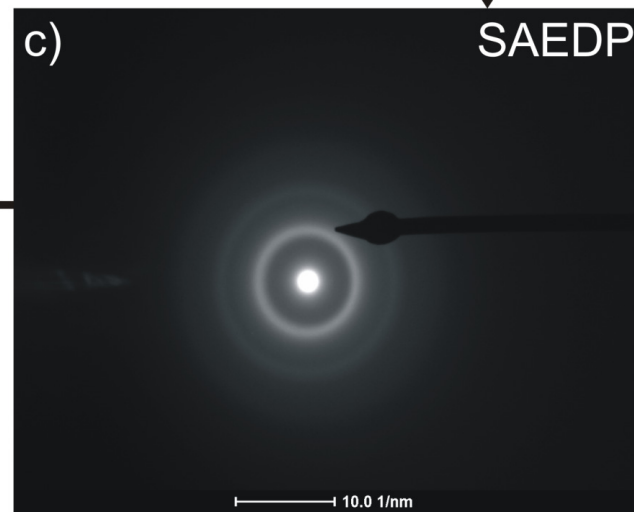
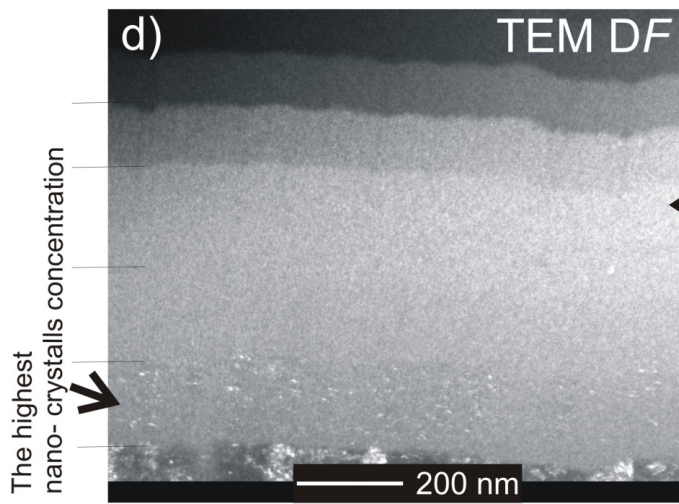
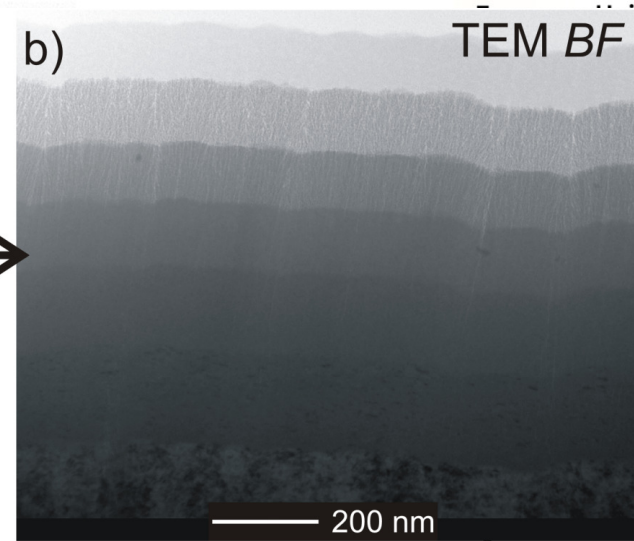
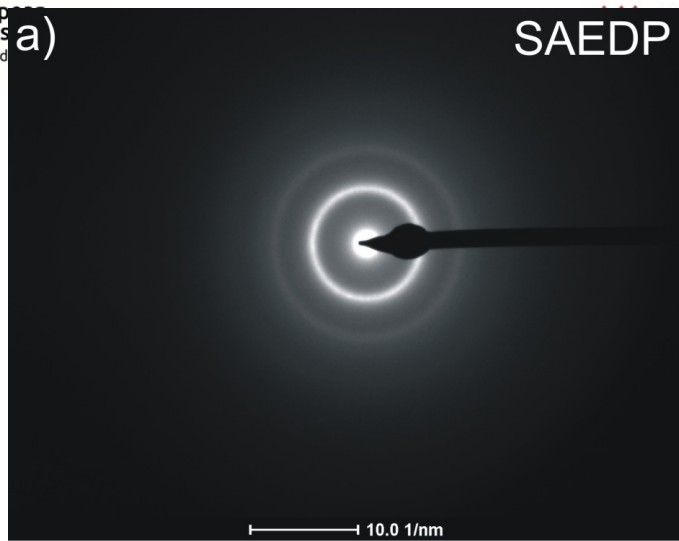




Off-axis Dark Field



On-axis Dark Field



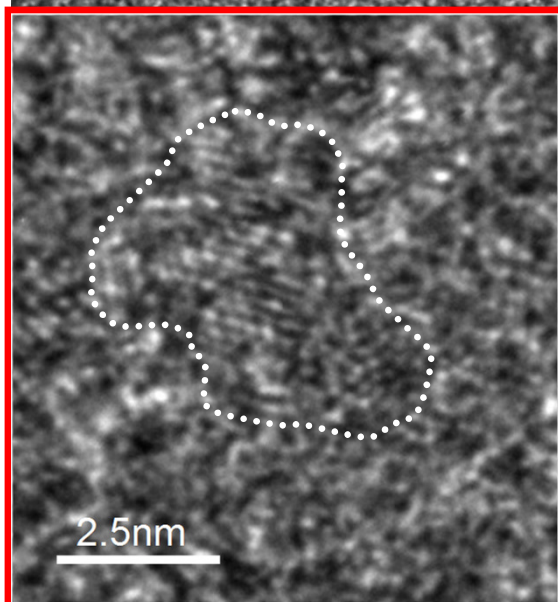


HRTEM

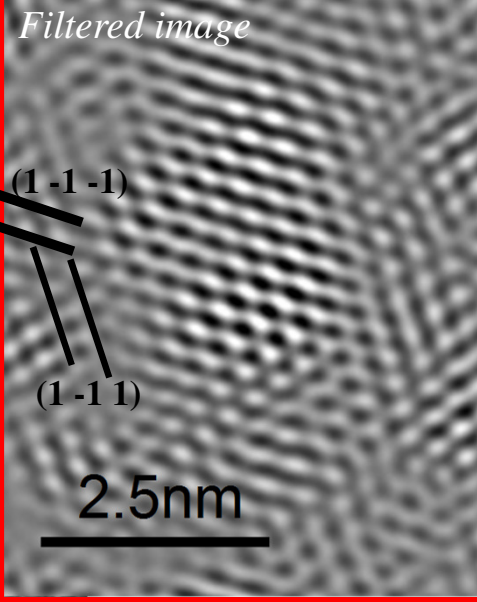


$(a:C-H + Cr)_A$

Zone axis $[1\ 1\ 0]$
 $Cr_{23}C_6$ hexagonal



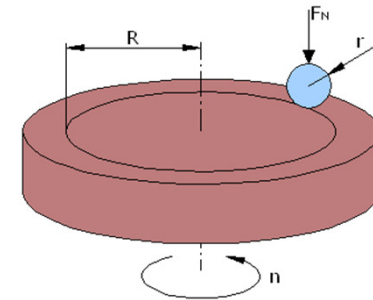
$(a:C-H + Cr)_B$





Tribological test– ball- on- disc (EN 1071-3:2003.)

Parameter:
indenter – Al_2O_3 ball, $d=1$ i 6mm ,



Low stress state

Load $F_N=1\text{N}$
Ball diameter $R=3\text{mm}$
Cycles number $n= 20.000$
Friction radius $r=5\text{mm}$,
Linear speed $v=0,06\text{m/s}$

$$\sigma_H=0,45 \text{ GPa}$$

High stress state

Load $F_N=5\text{N}$
Ball diameter $R=3\text{mm}$
Cycles number $n= 5.000$
Friction radius $r=4\text{mm}$,
Linear speed $v=0,05\text{m/s}$

$$\sigma_H=0,8 \text{ GPa}$$



European
SEM

1N- test

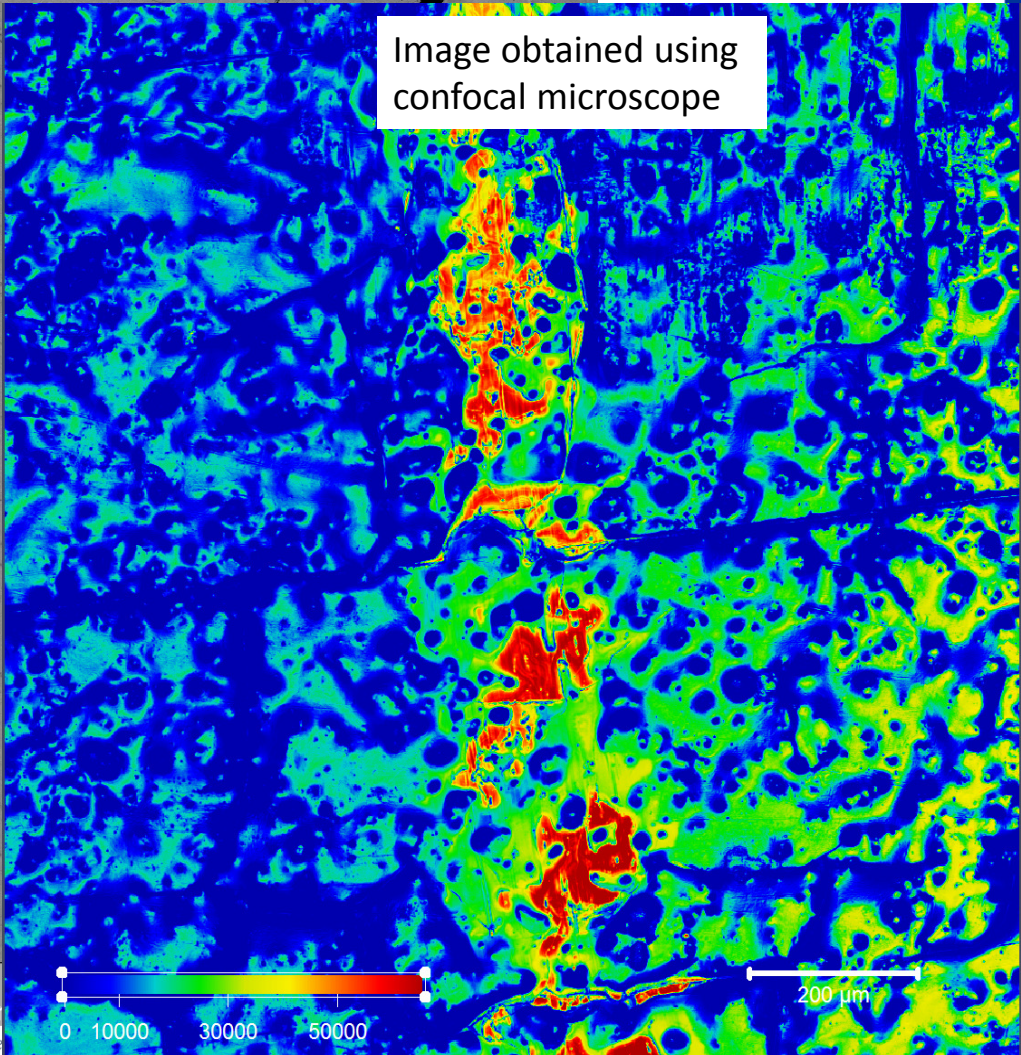
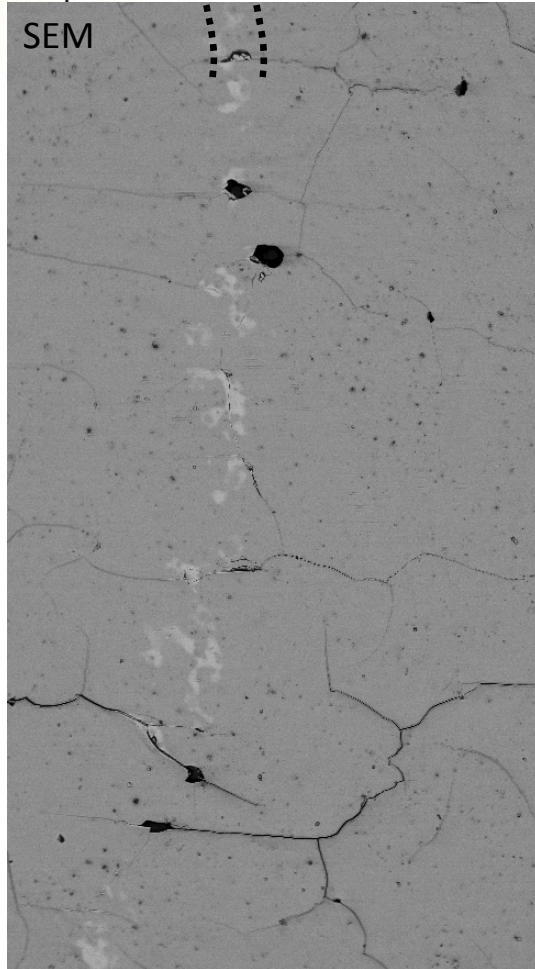
INSTITUTE OF METALLURGY

5N- test

European Union



Image obtained using
confocal microscope



11/28/2013	HV	mag	WD
5:33 AM	30.00 kV	50 x	14.8 mm



International inte

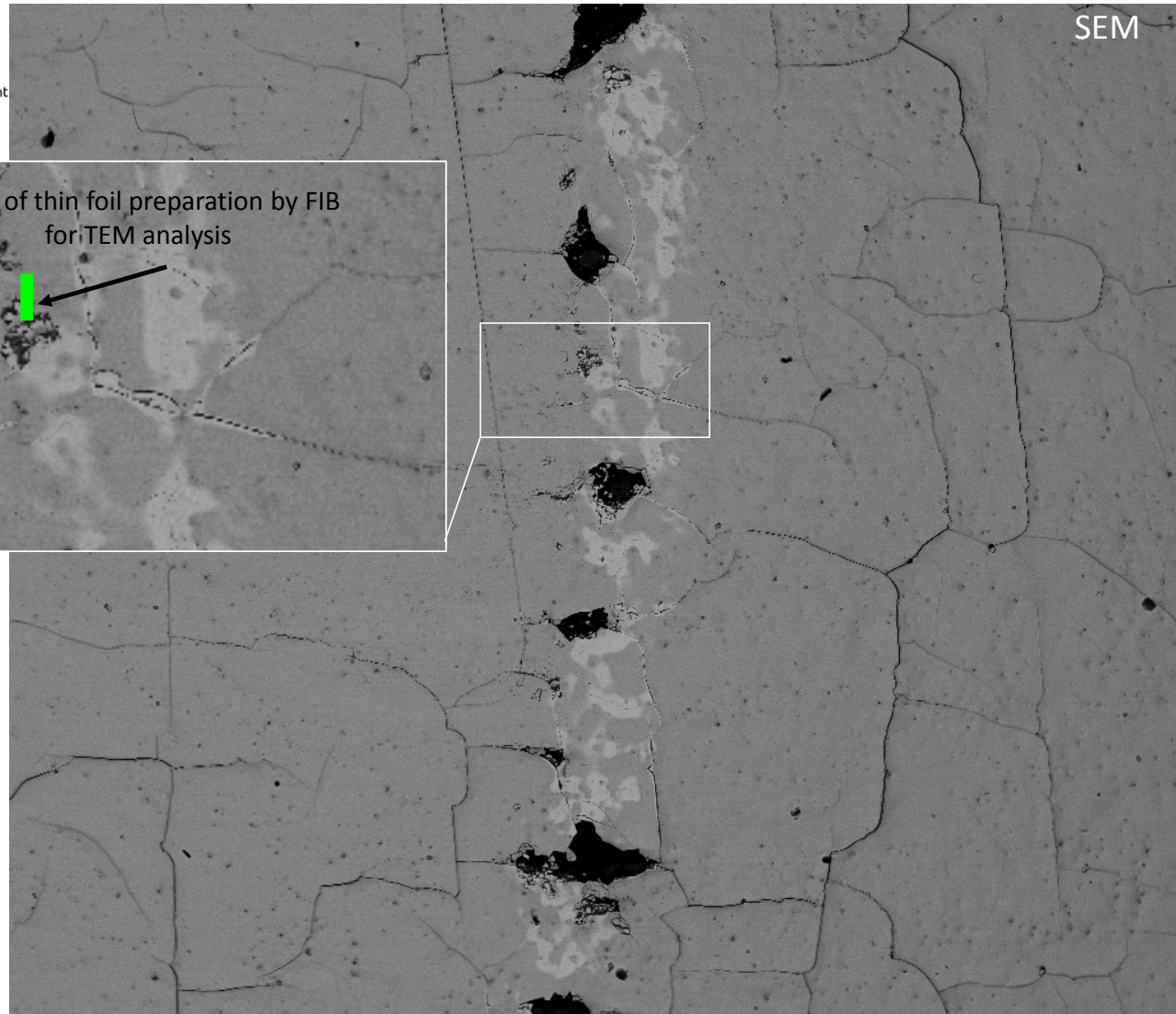
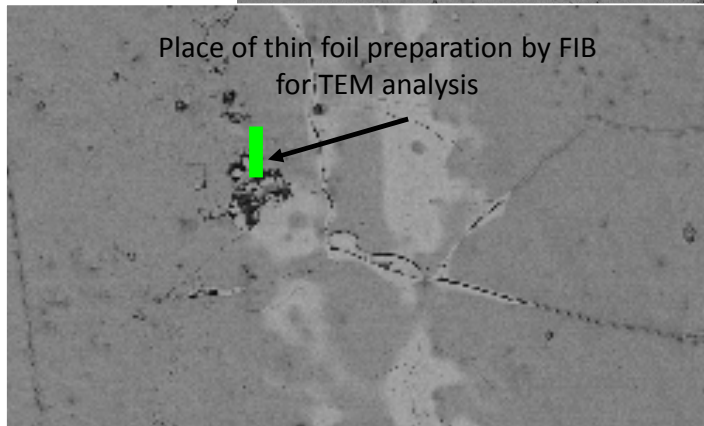
Project co-financed by the European Union within the European Social Funds




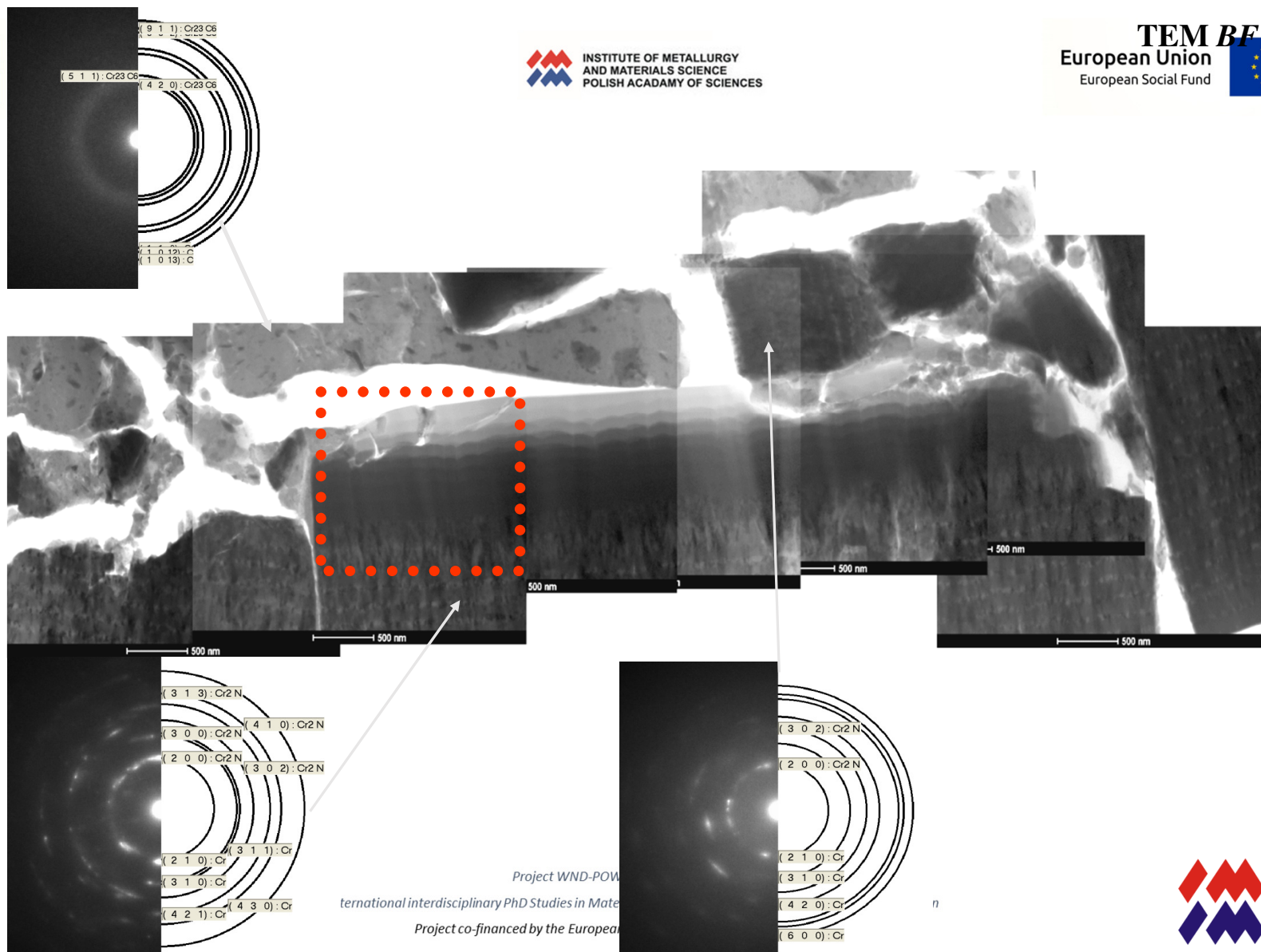
European Funds
Knowledge Education Development



SEM



	11/28/2013 10:59:45 AM	HV 30.00 kV	mag 50 x	WD 14.8 mm	HFV 2.70 mm	1 mm	Quanta 3D
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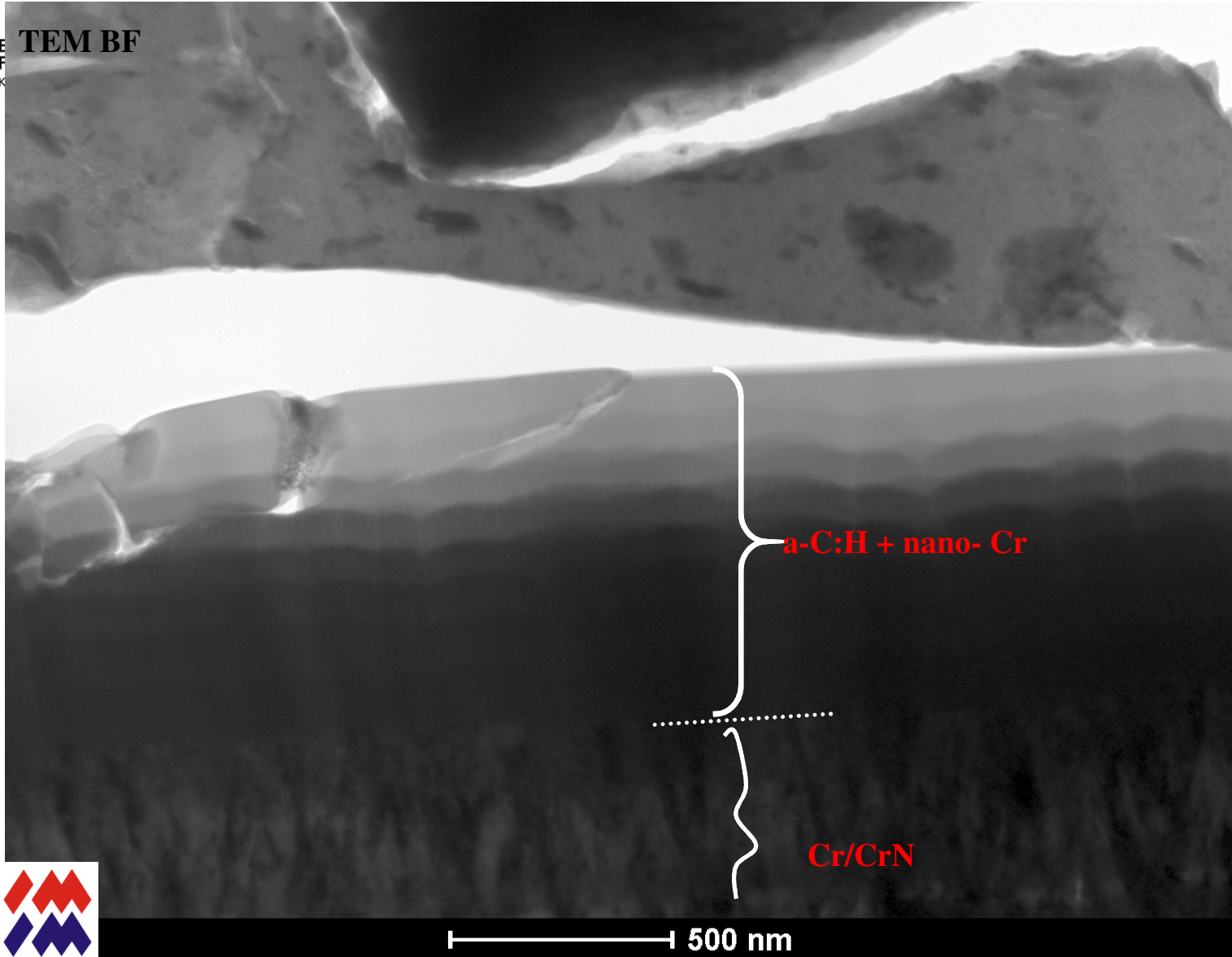


Project WND-POW
International interdisciplinary PhD Studies in Materials Science
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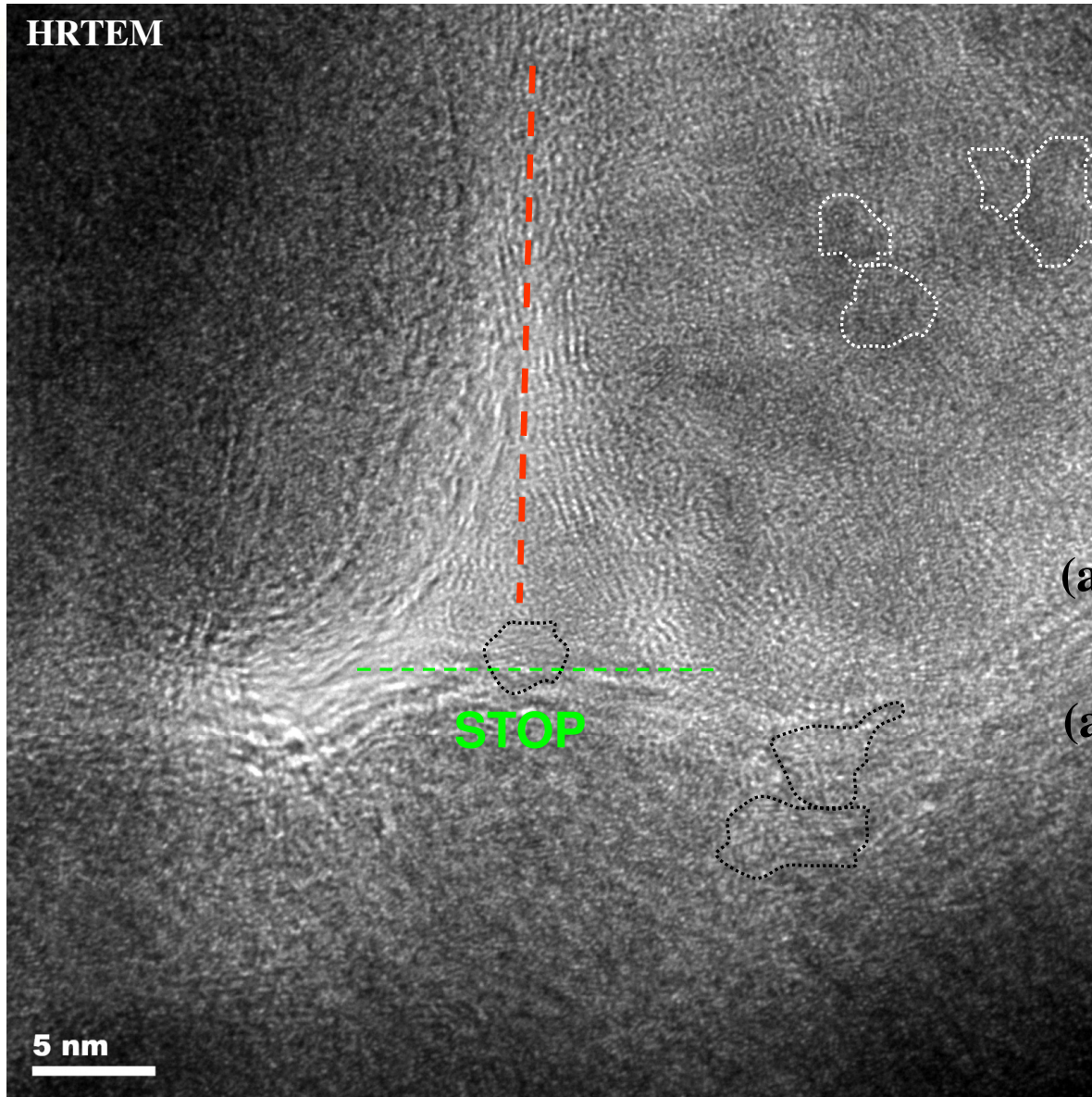
TEM BF



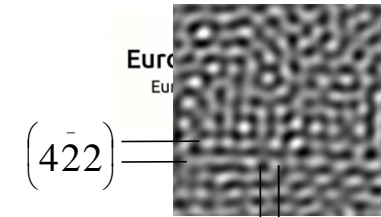
500 nm



HRTEM



5 nm



Cr_{23}C_6 cubic-
Zone axis $[\bar{2}15]$

(a-C:H+ nano Cr)_A

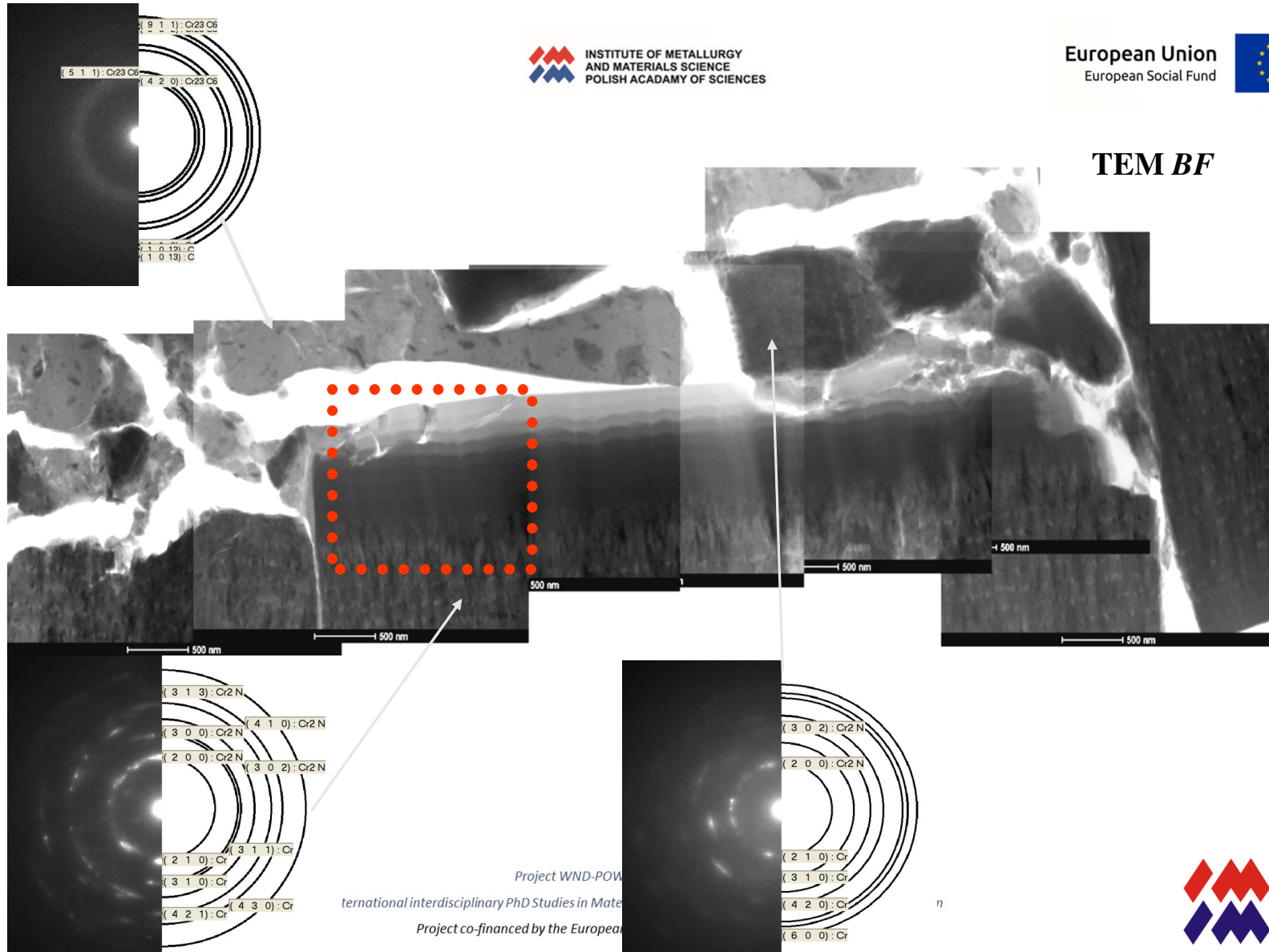
.....Interface

(a-C:H+ nano Cr)_B





TEM BF

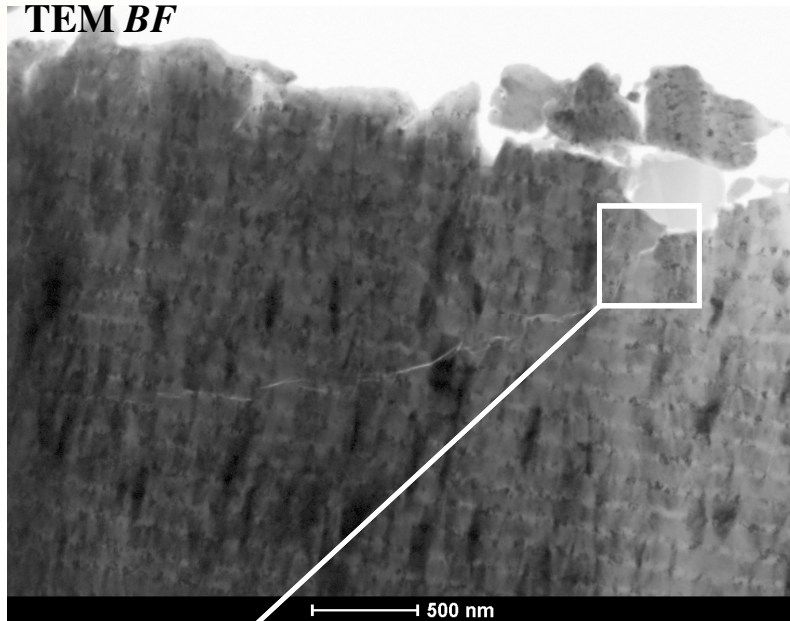


Project WND-POW
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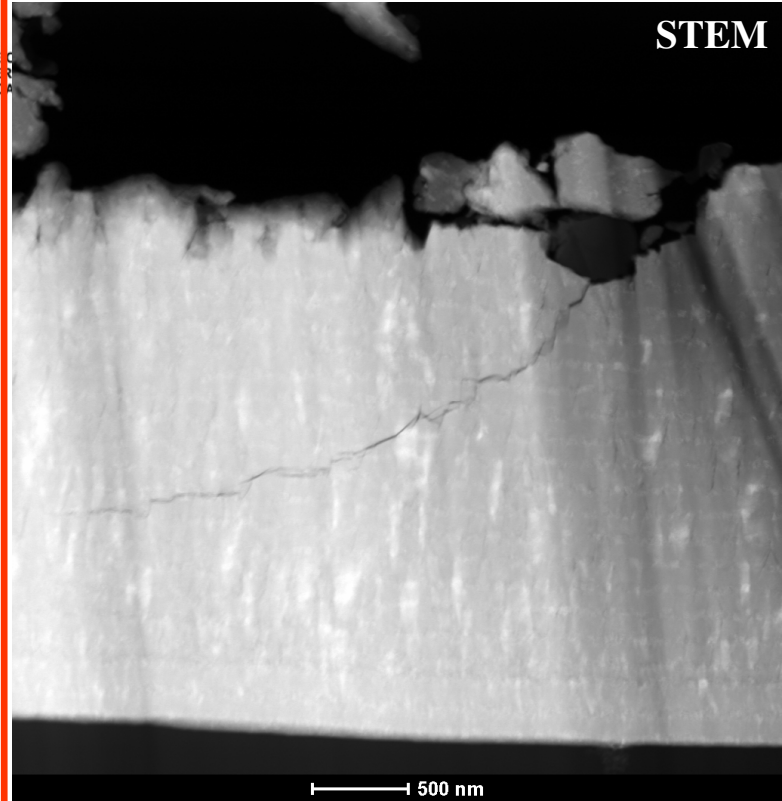




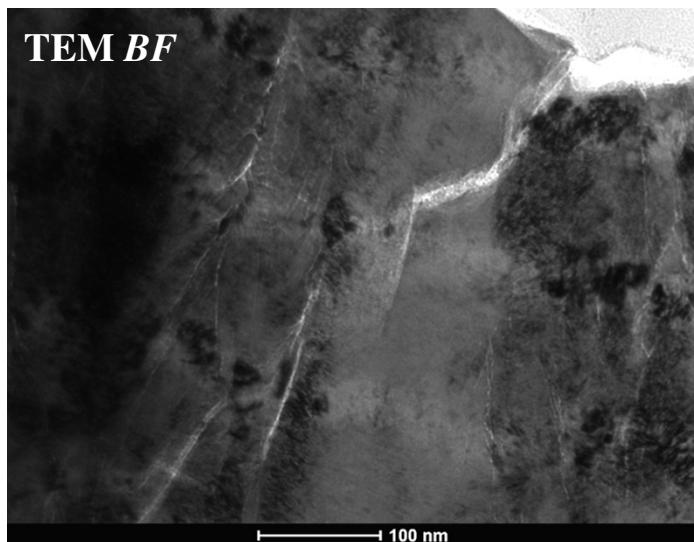
TEM BF



STEM



TEM BF



WND-POWR.03.02.00-00-1043/16

es in Material Science with English as the language of instruction

European Union within the European Social Funds





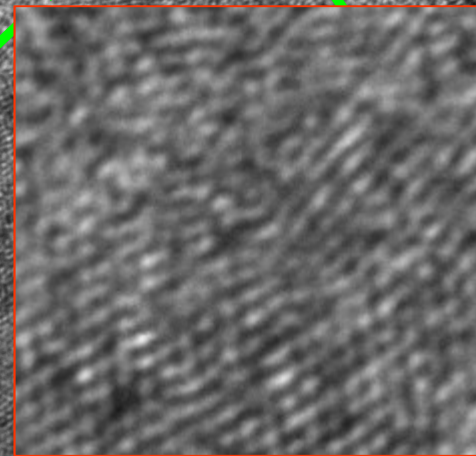
Cr_2N

Interface

Cr

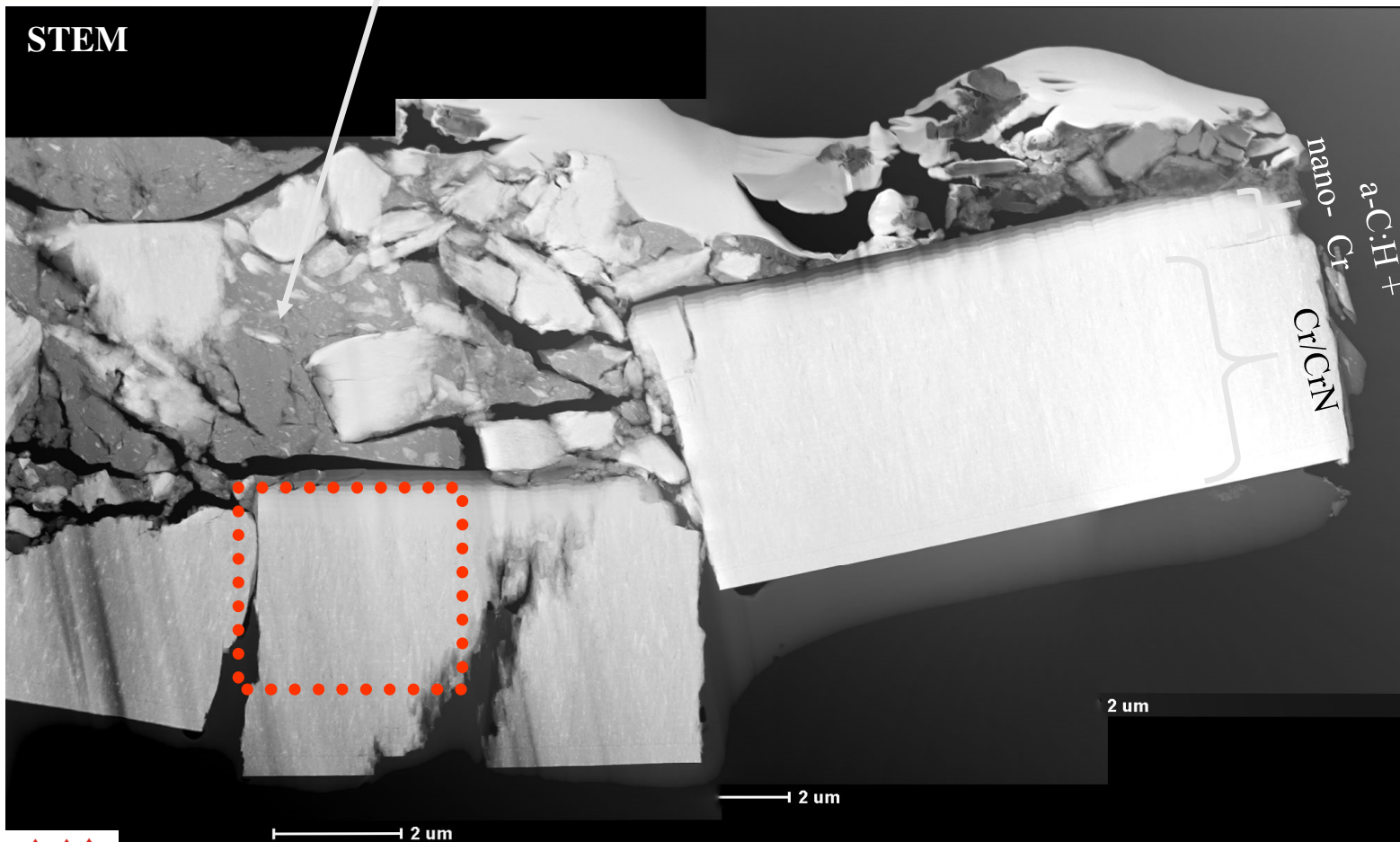
5 nm

45°





Tribo- film



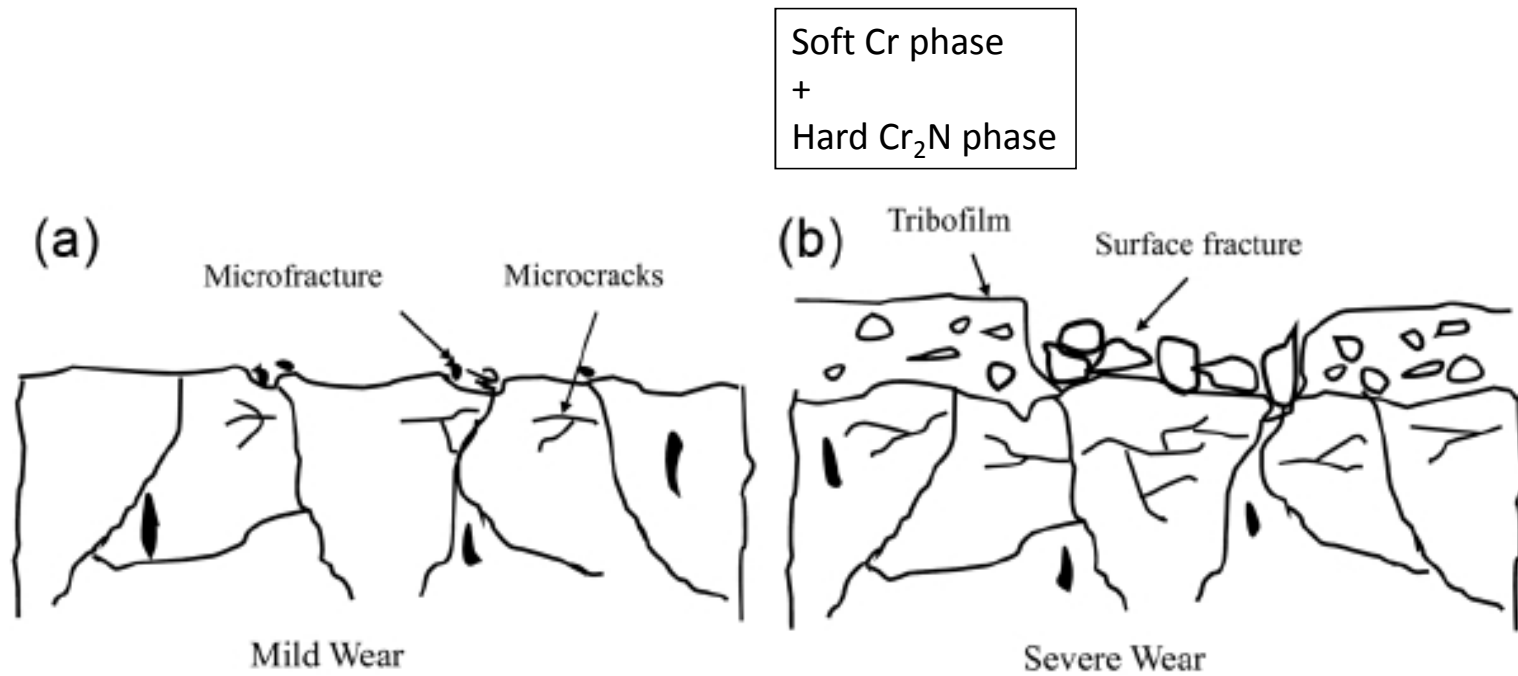
Project WND-POWR.03.02.00-00-1043/16

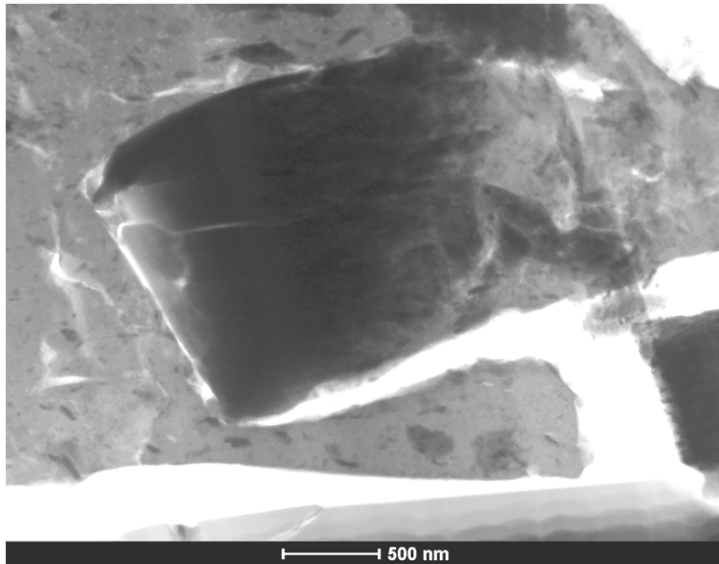
International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

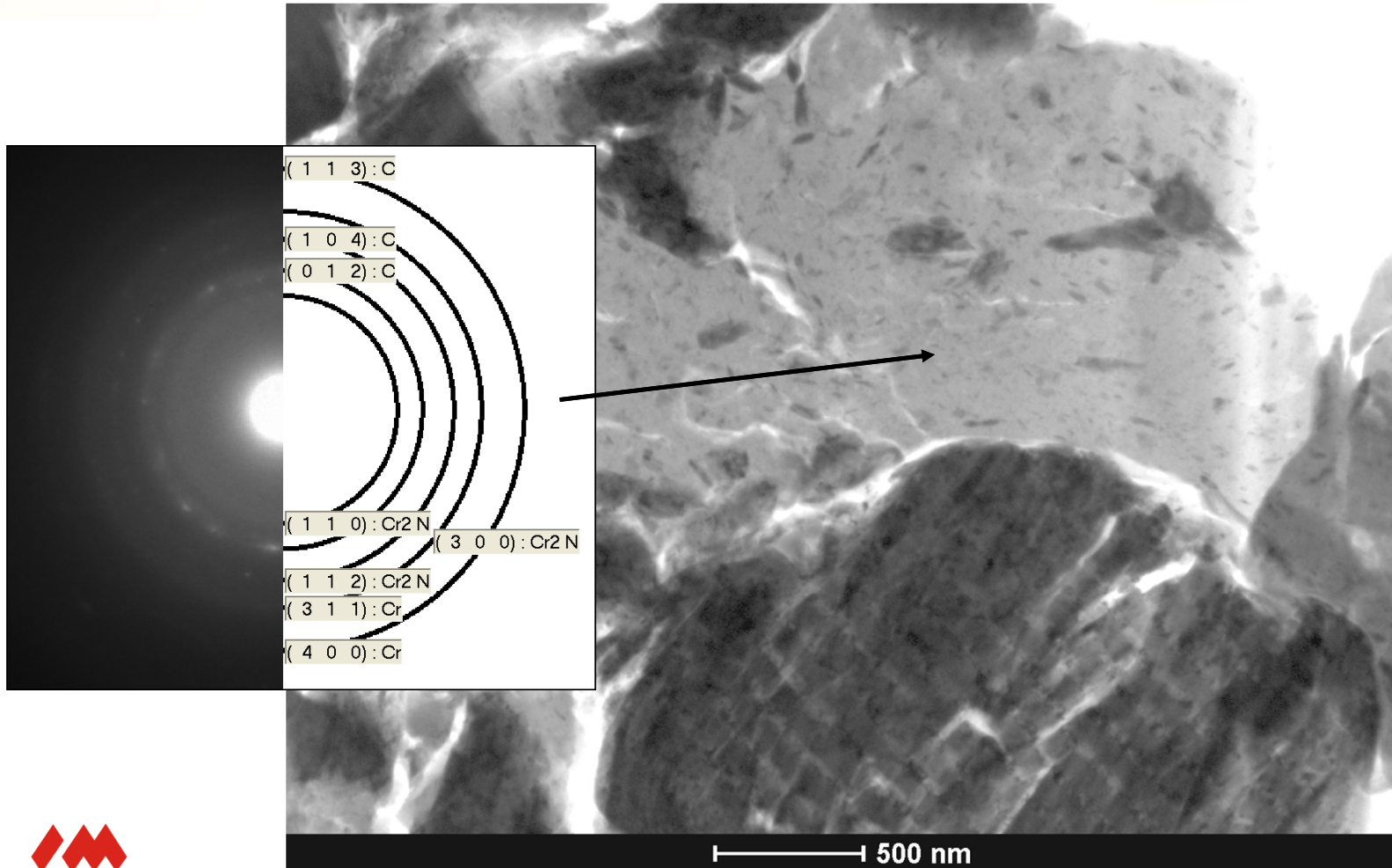
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Microstructural characterization of the coatings by TEM after the wear test









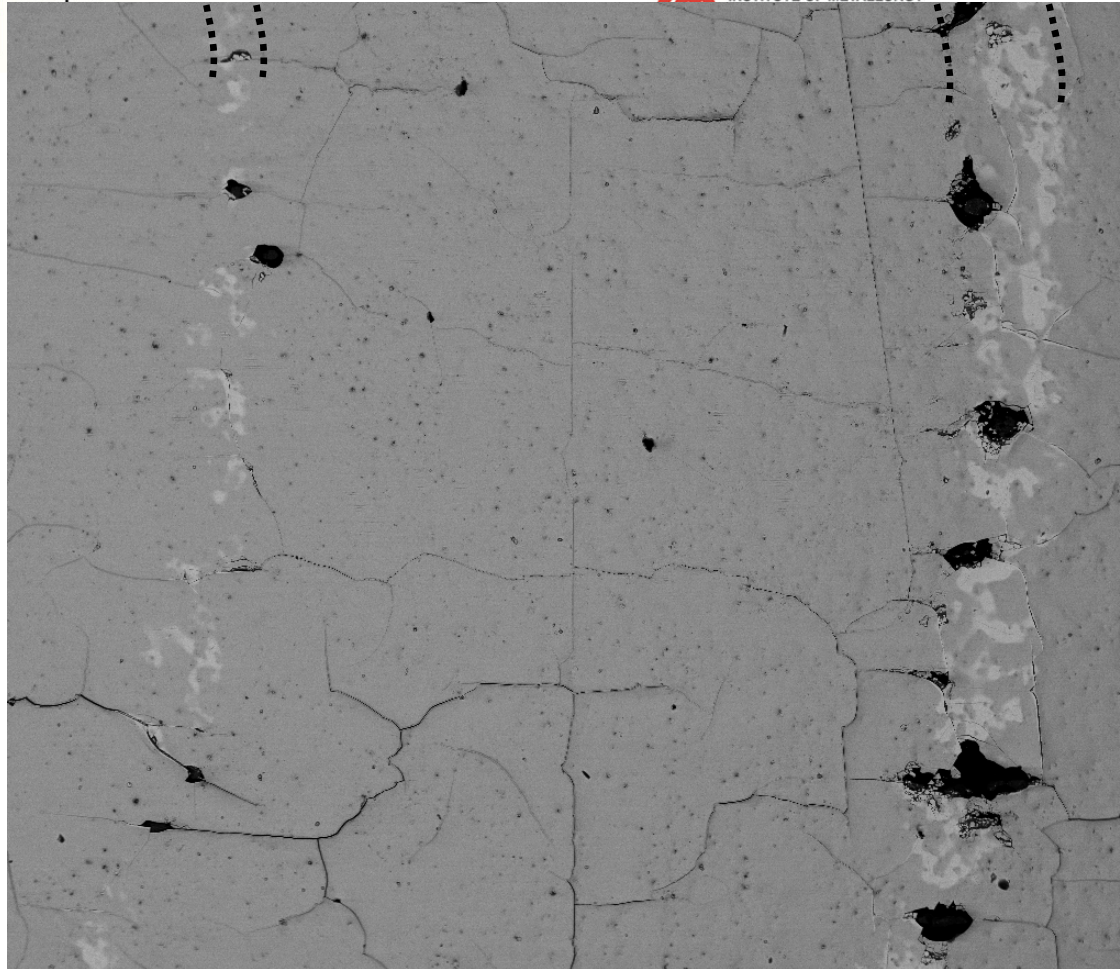
European



1N- test

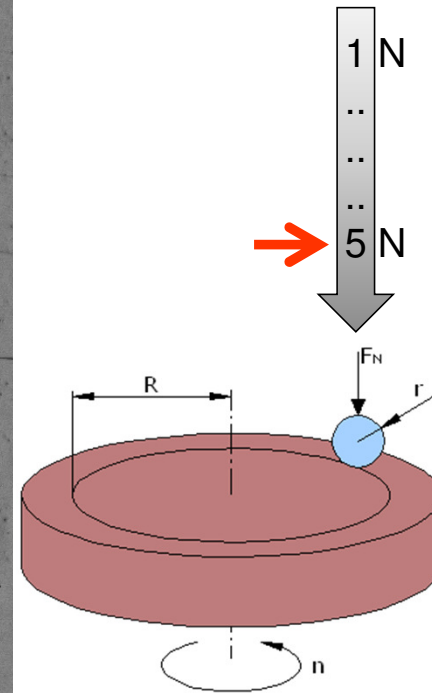
INSTITUTE OF METALLURGY

5N- test



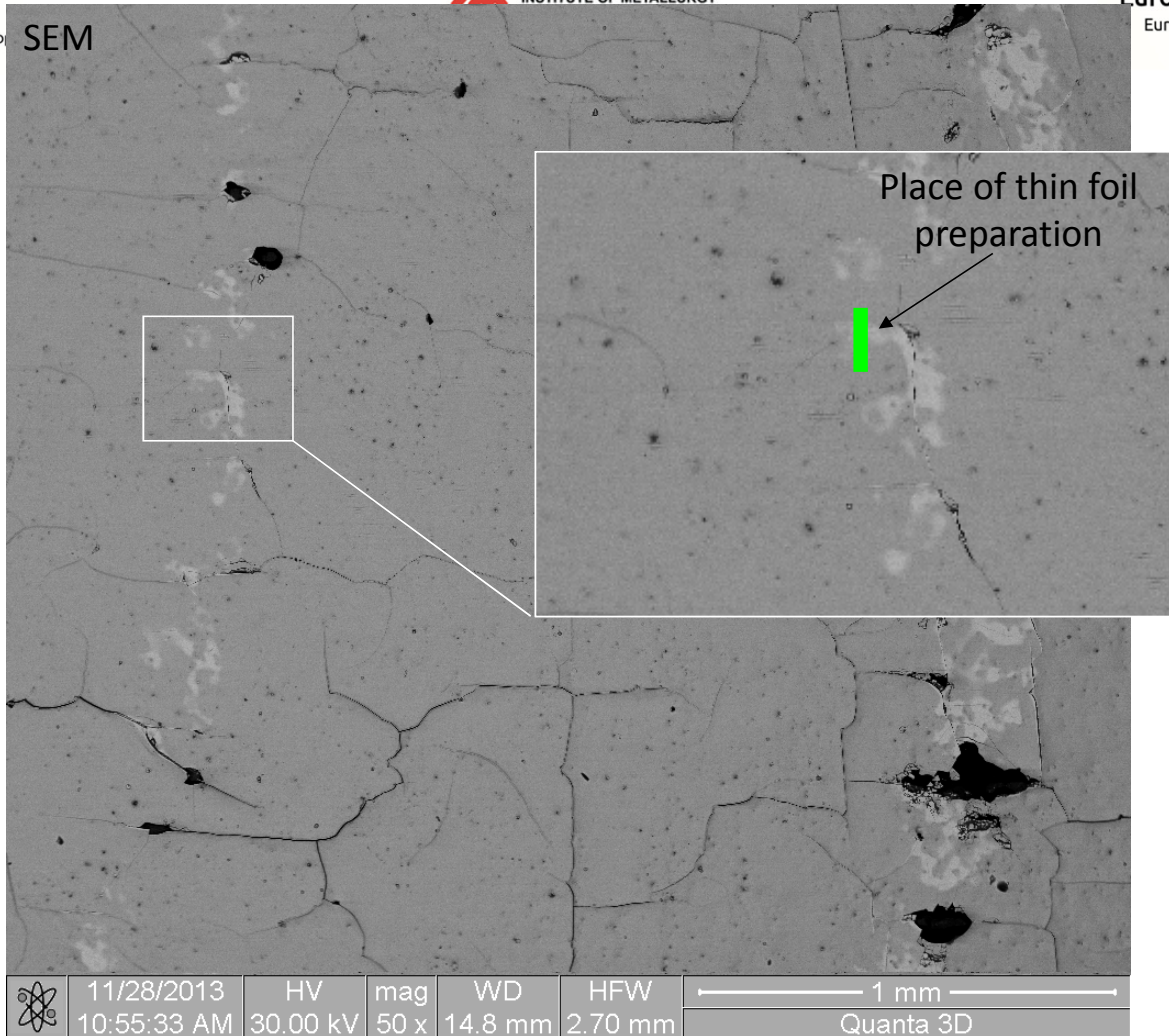
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						Quanta 3D

SEM
European Union
European Social Fund



International interdisciplinary PhD Studies in Materials Science with English as the language of instruction
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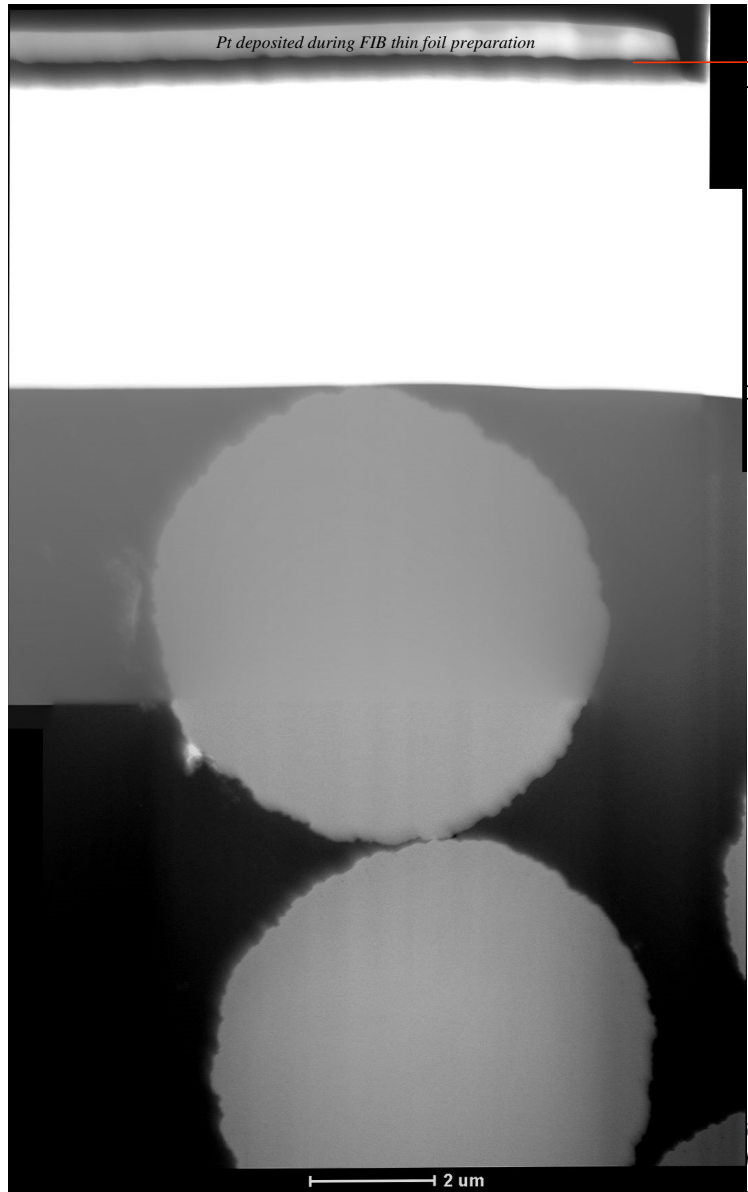


International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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STEM



a-C:H + Cr nano

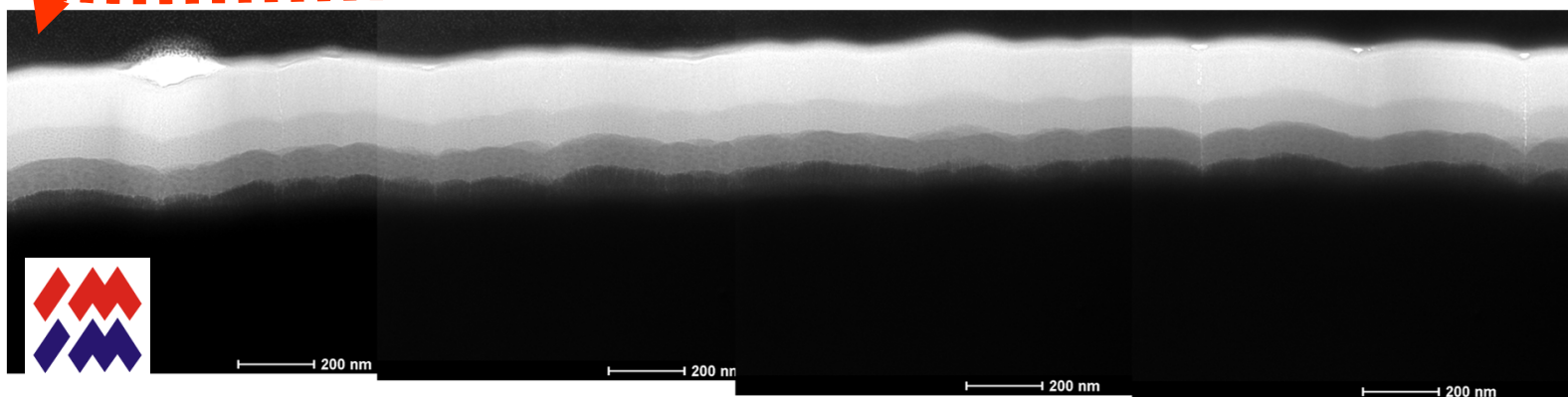
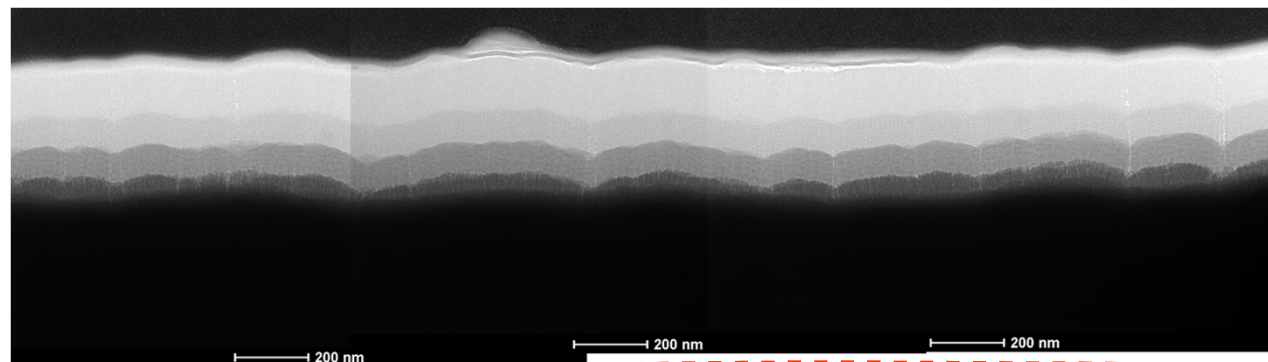
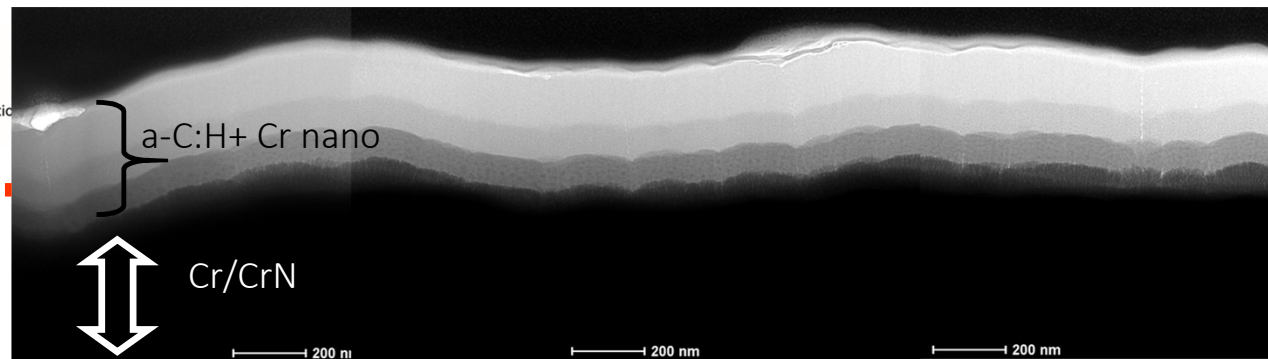
Cr/CrN

substrate CFC

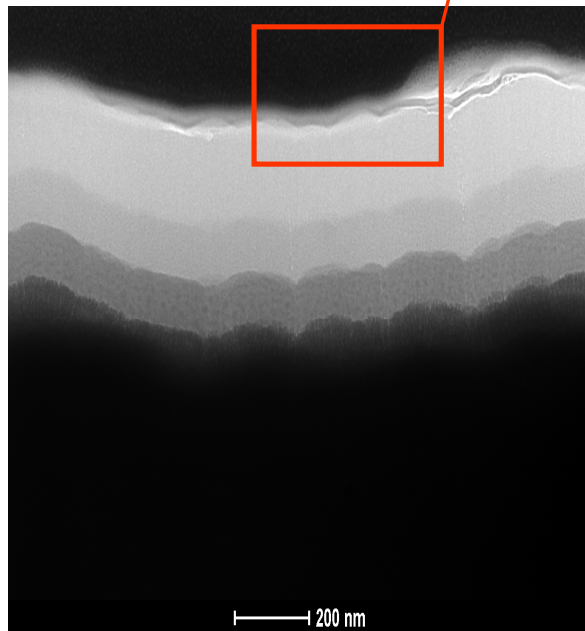
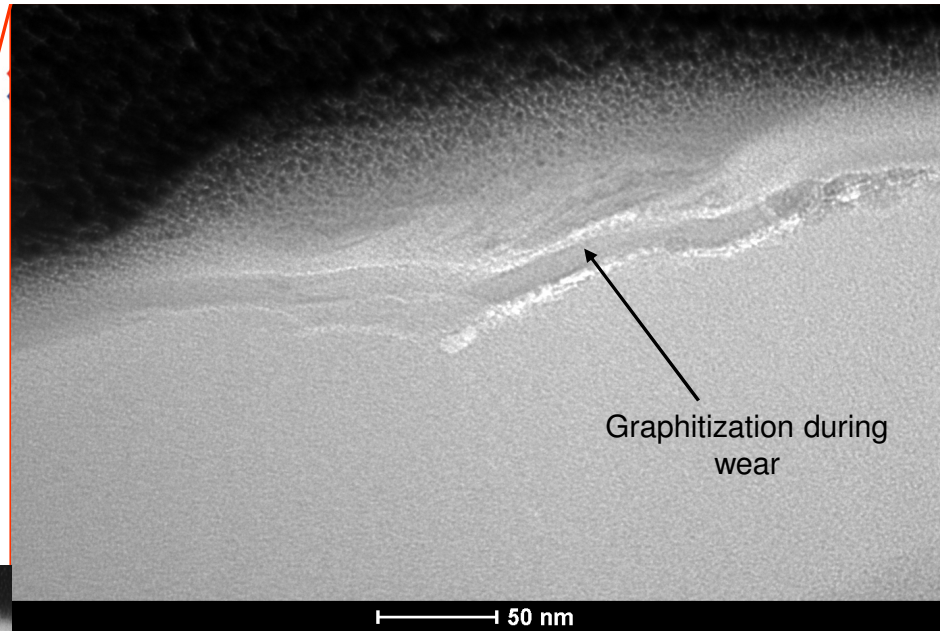




European Union
Social Fund



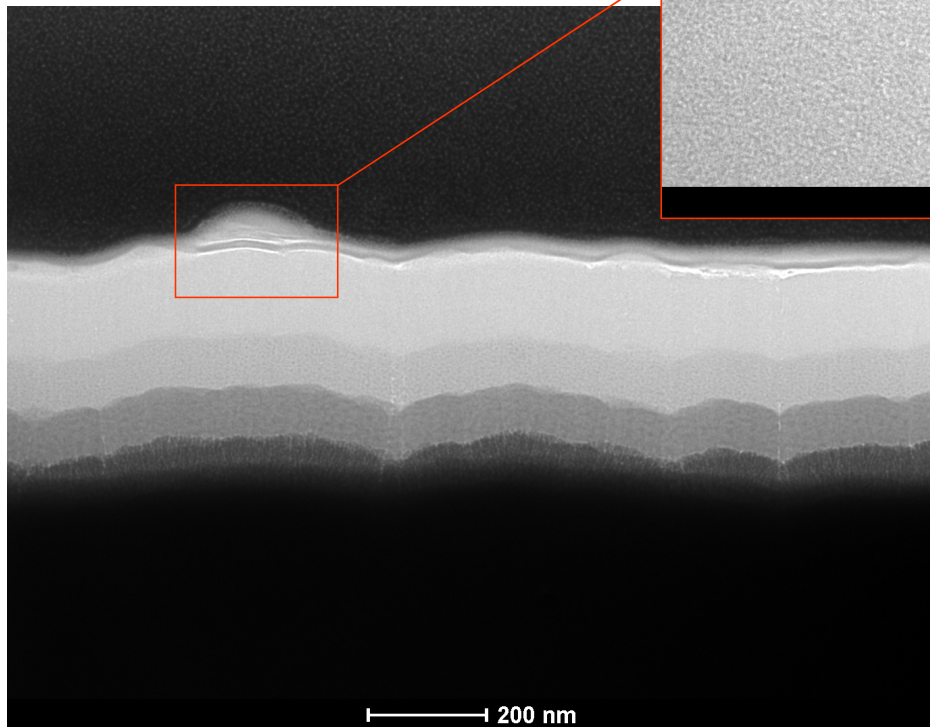
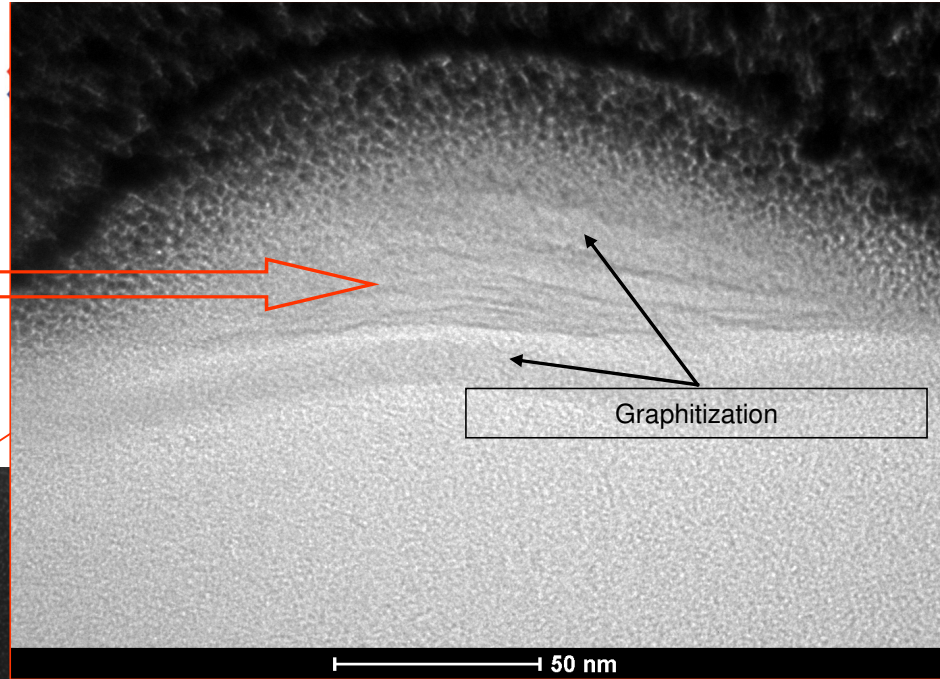
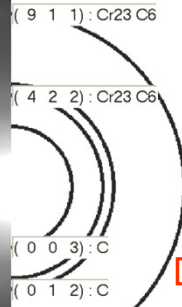
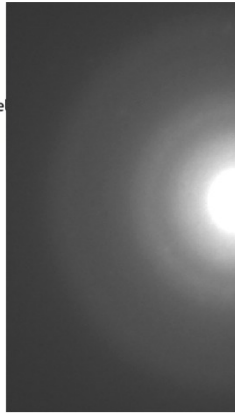
TEM
BF





European Funds
Knowledge Education Deve

TEM
BF +
SAED

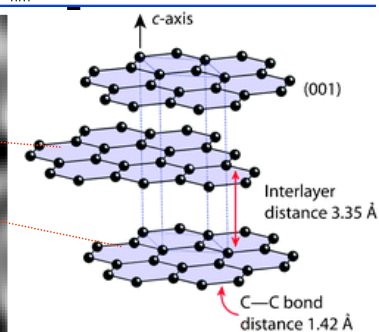
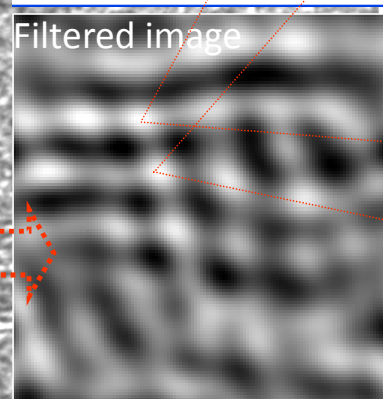
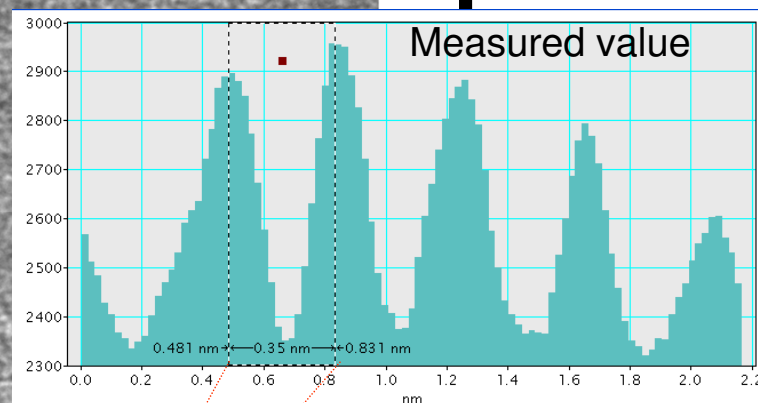
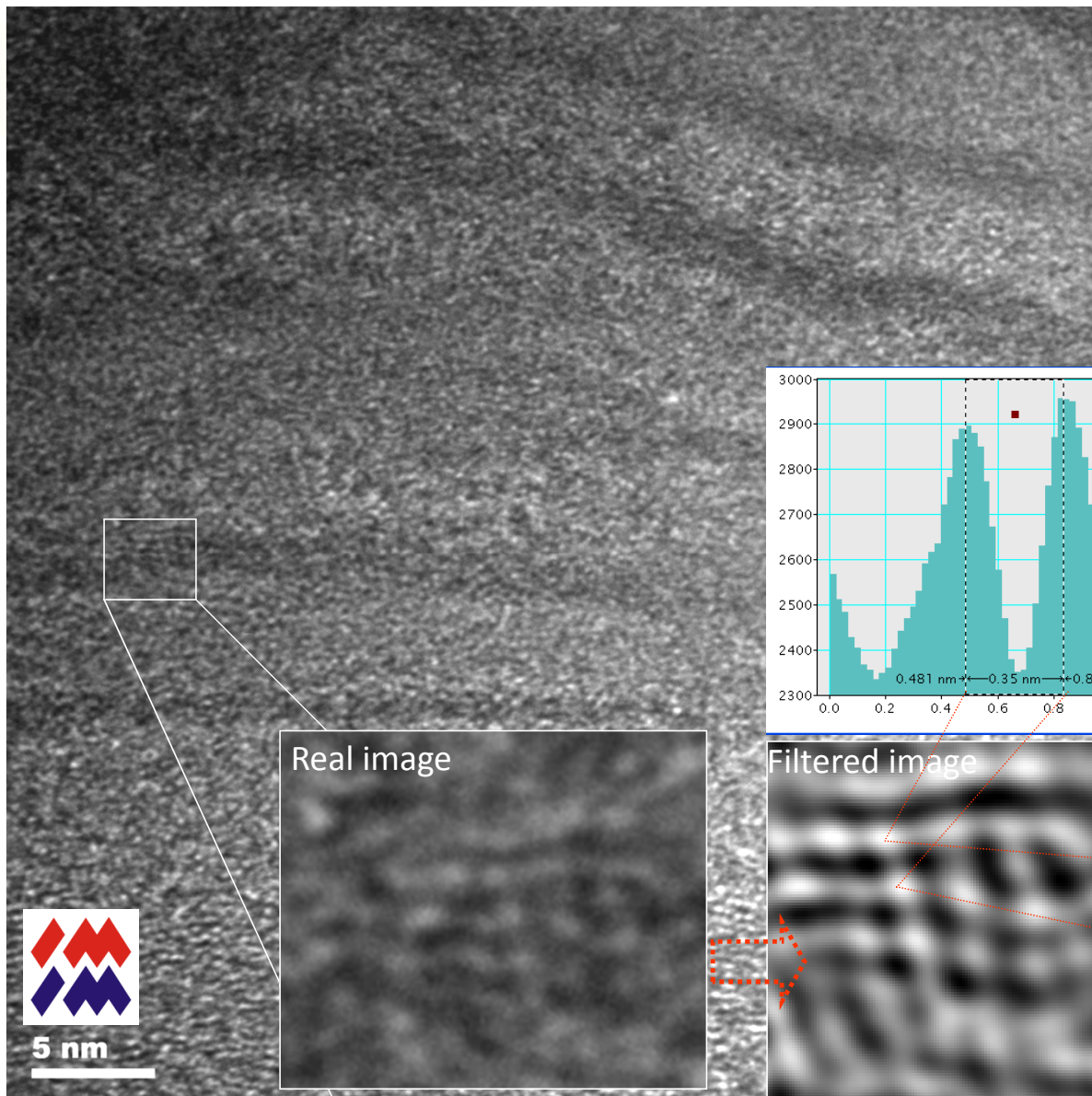




European Union
European Social Fund



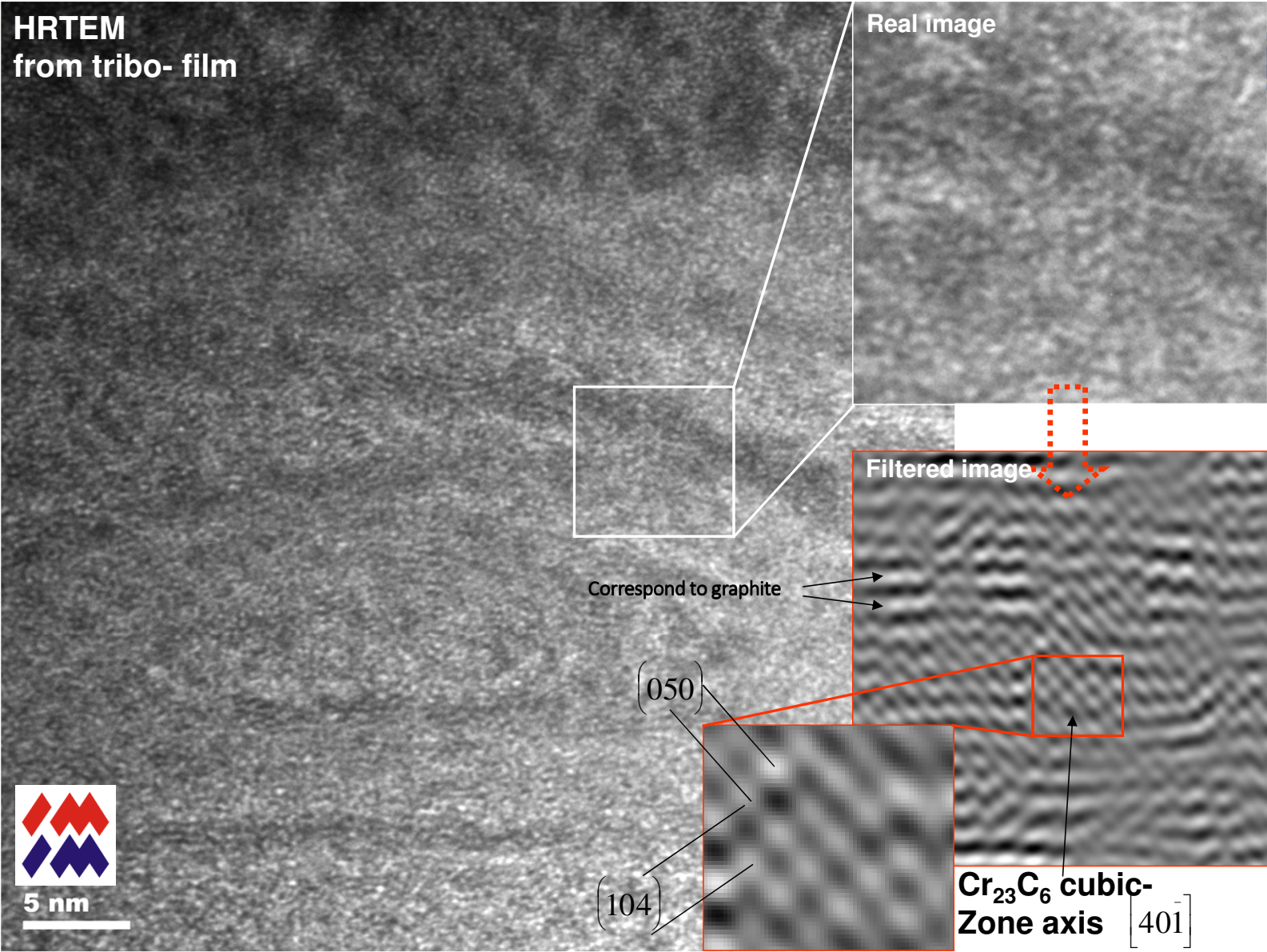
HRTEM graphite- Tribo-film



Theoretical value



HRTEM from tribo- film





- Project NCN nr: 3066/B/T02/2011/40- *FINISHED*
- Project NCN nr: 2012/06/M/ST8/00408- *HARMONIA- FINISHED*
- Project NCN nr: 2012/07/B/ST8/03396- *OPUS- FINISHED*
- Project NCN nr: 2014/15/B/ST8/00103- *OPUS- FINISHED*
- Project NCN nr: 2015/19/B/ST8/00942- *OPUS- in progress*
- Project NCBR, number: DZP/M-ERA.NET-2015/285/2016- *in progress*



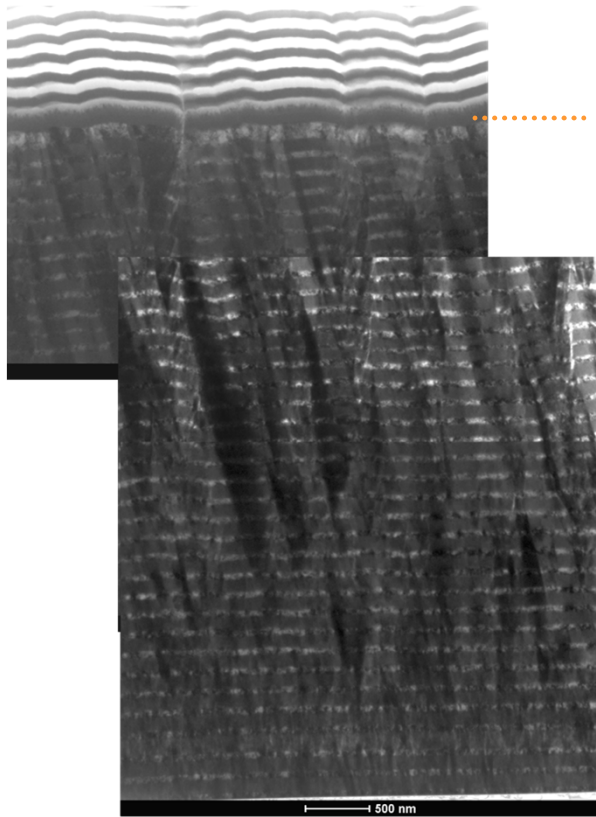
Title: Biomechanical and microstructure analysis of multilayer- nano-composite protective coatings on metallic substrates for tissue interaction

Project WND-POWR.03.02.00-00-1043/16

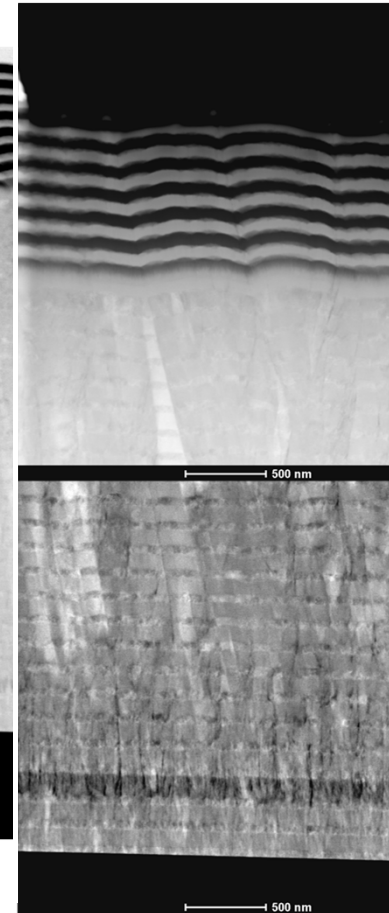
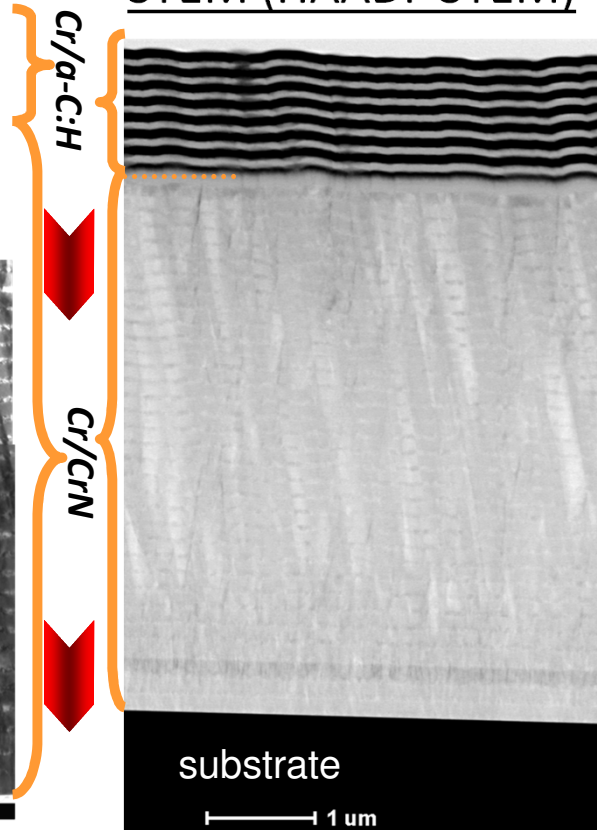
International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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TEM BF



STEM (HAADF STEM)

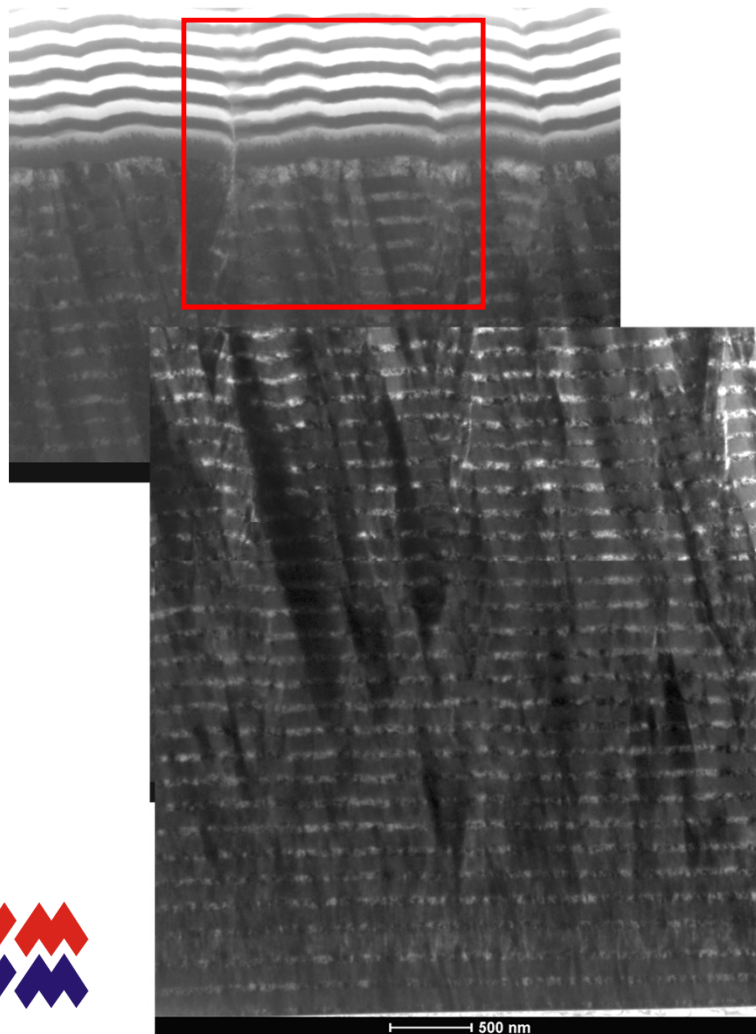


Project WND-POWR.03.02.00-00-1043/16

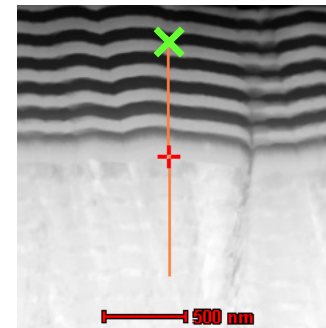
International interdisciplinary PhD Studies in Materials Science with English as the language of instruction
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TEM BF



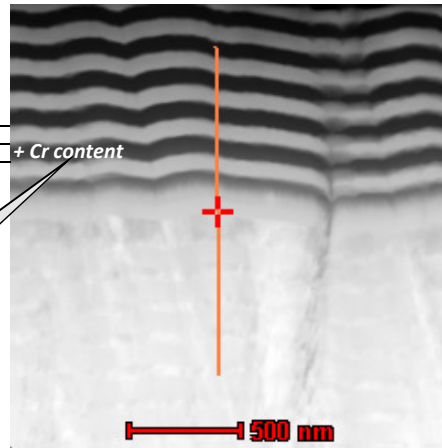
STEM (HAADF STEM)



02.00-00-043/16

science with English as the language of instruction

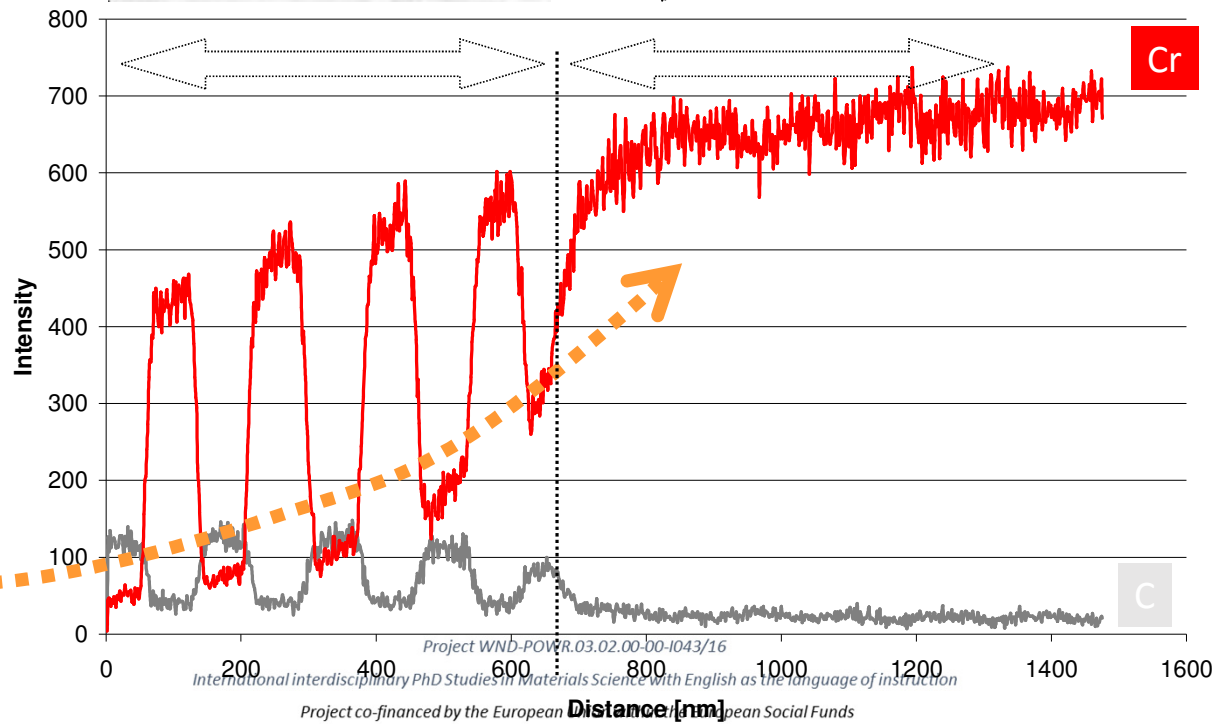
within the European Social Funds



Cr/ a-C:H

Cr/ CrN

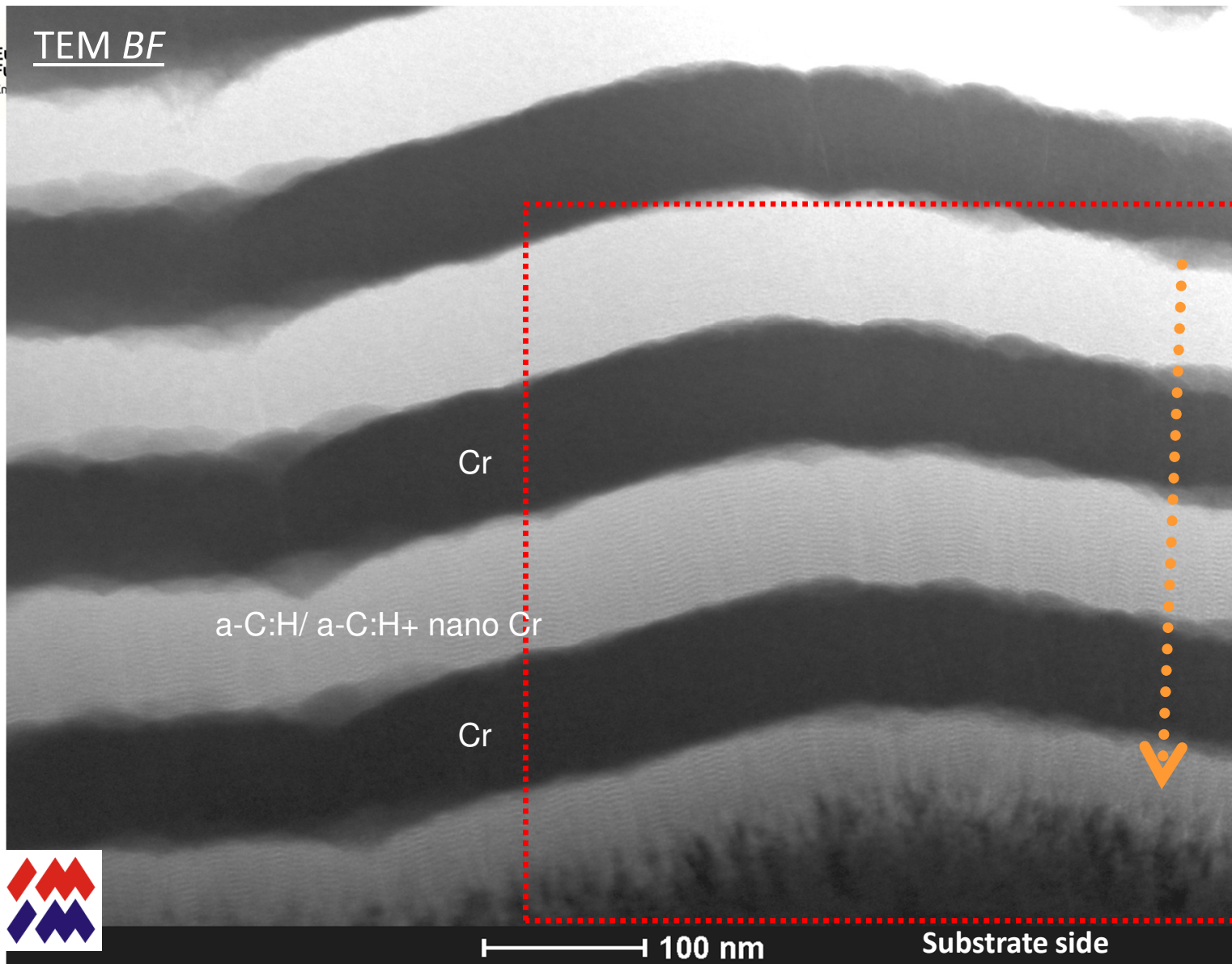
Question:
How Cr
is distributed
in a-C:H areas ?

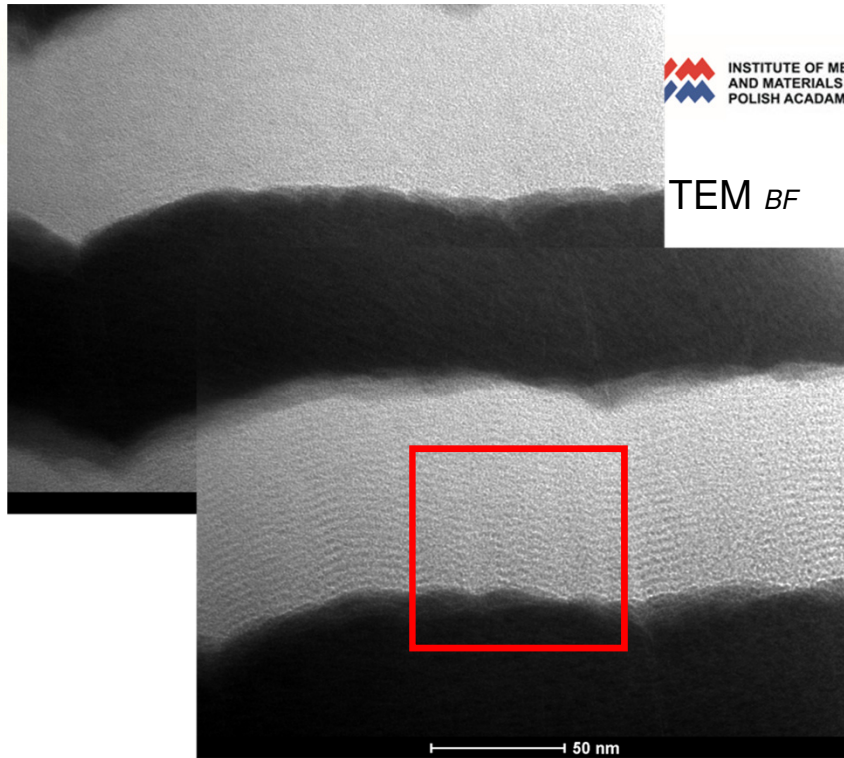




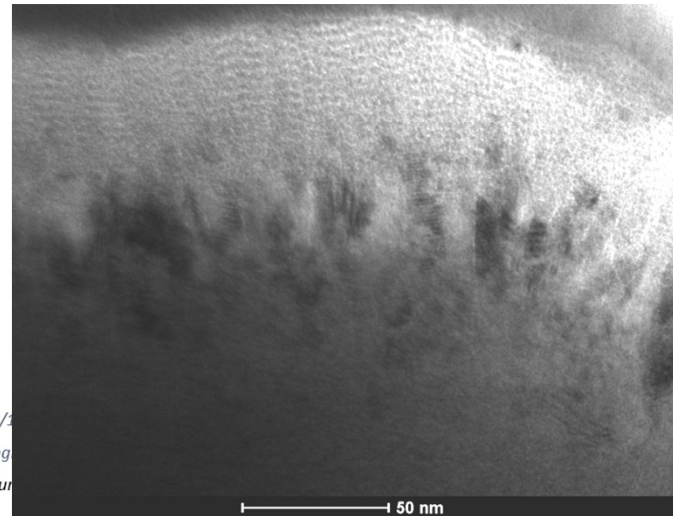
Erasmus
F
Kn

TEM BF



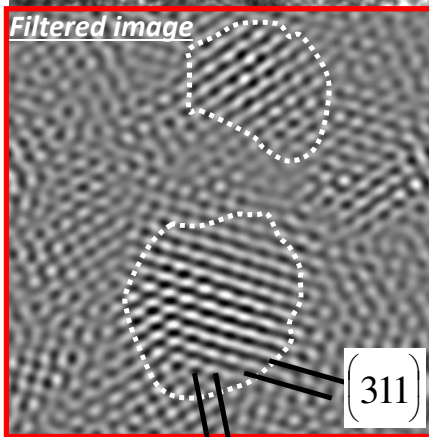
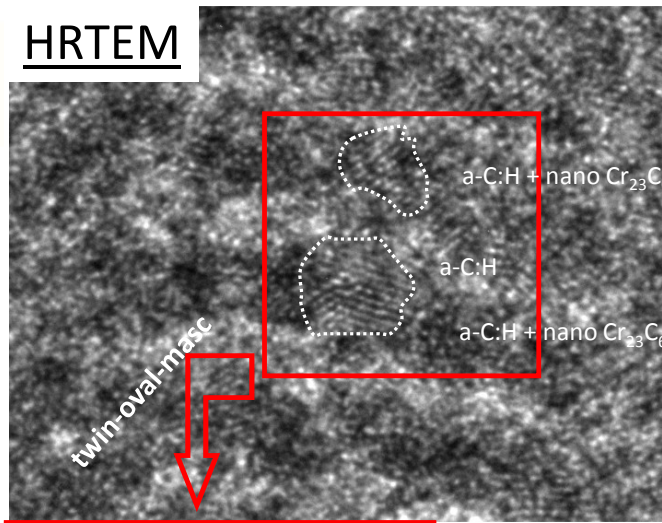


a-C:H/a-C:H+nano Cr





HRTEM



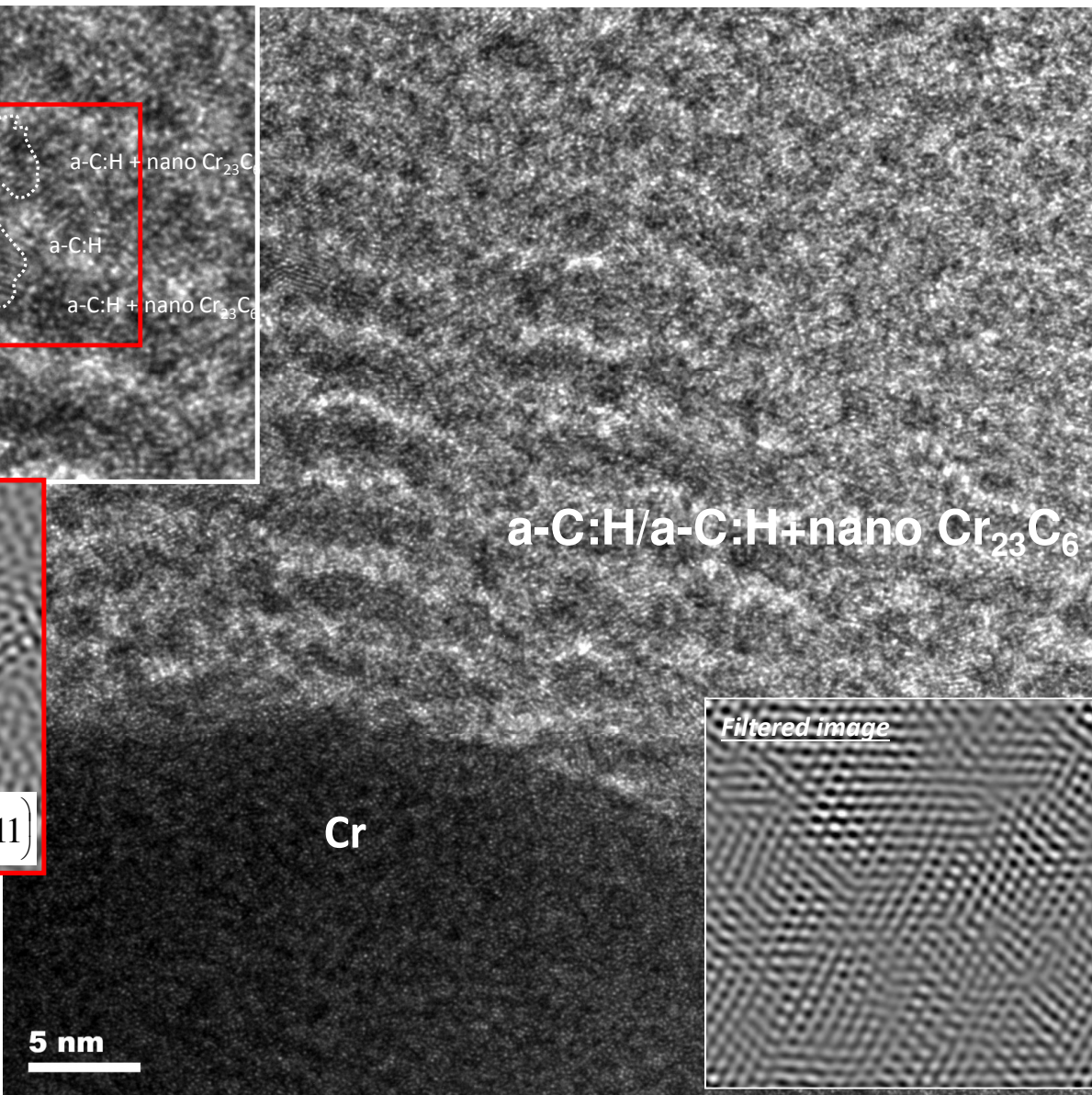
Zone axis $\bar{4}93$
 $Cr_{23}C_6$



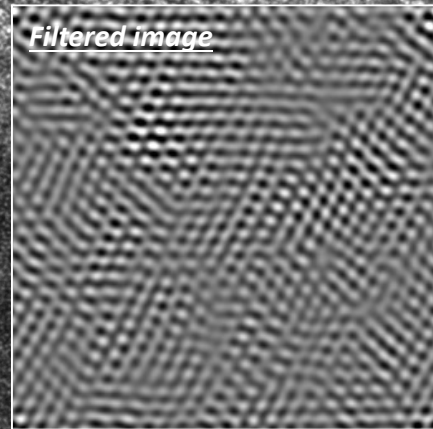
(322)

(311)

5 nm



a-C:H/a-C:H+nano $Cr_{23}C_6$





European Union

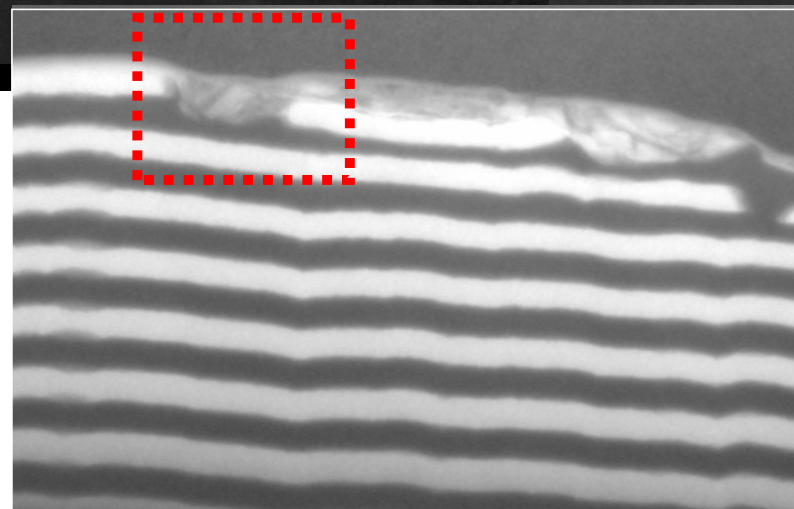
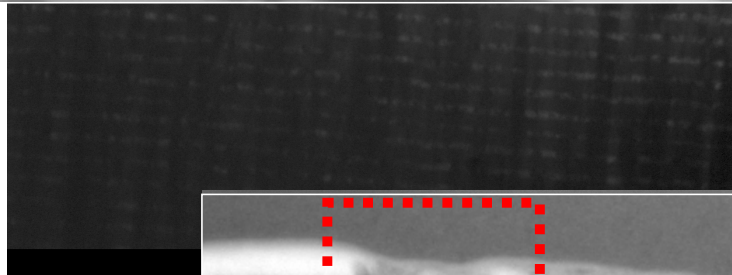
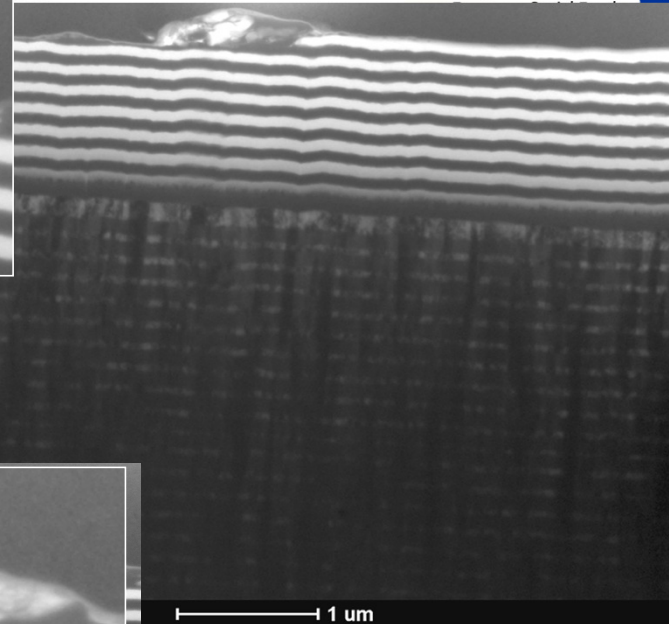
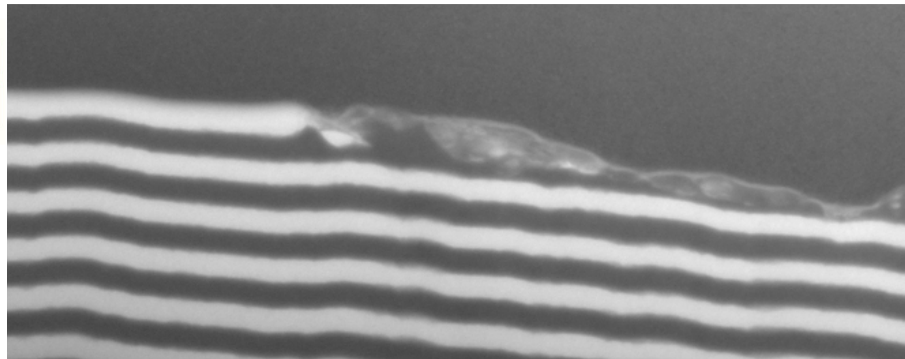
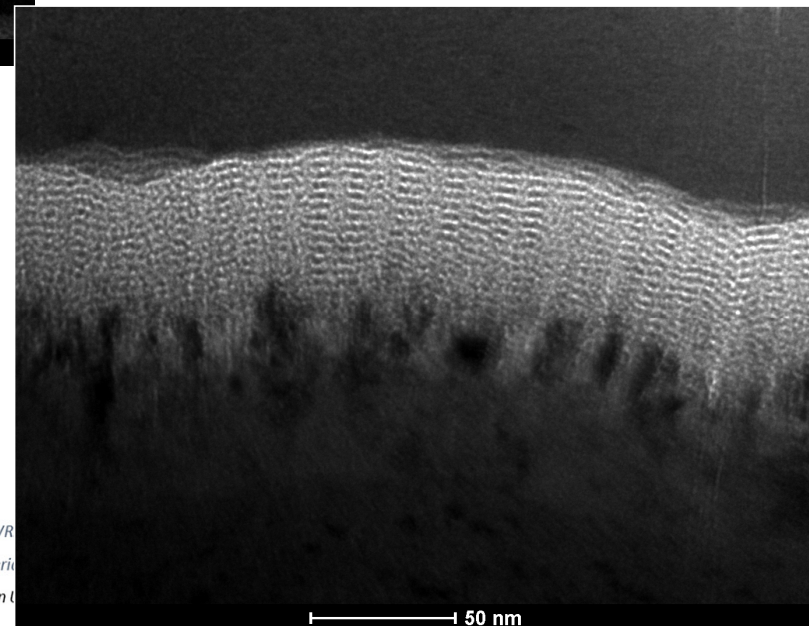
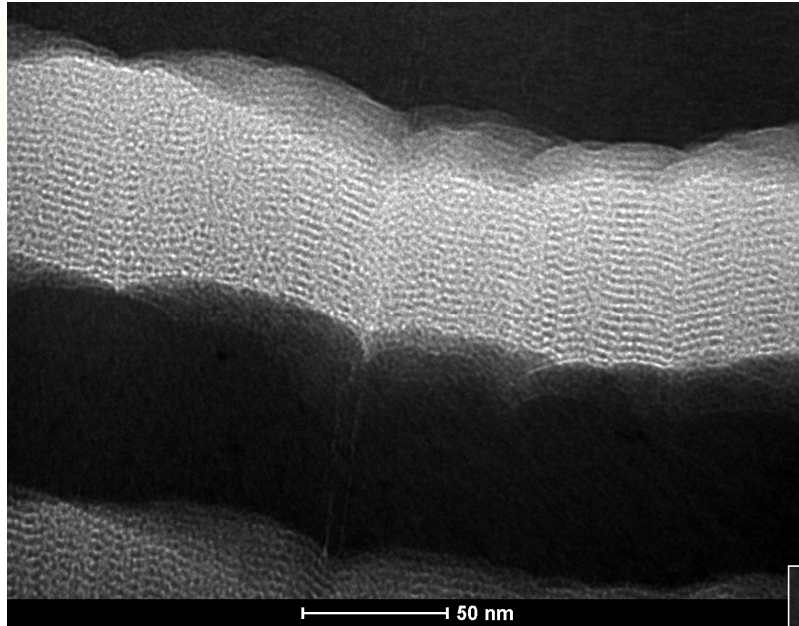


Image of instruction
ends

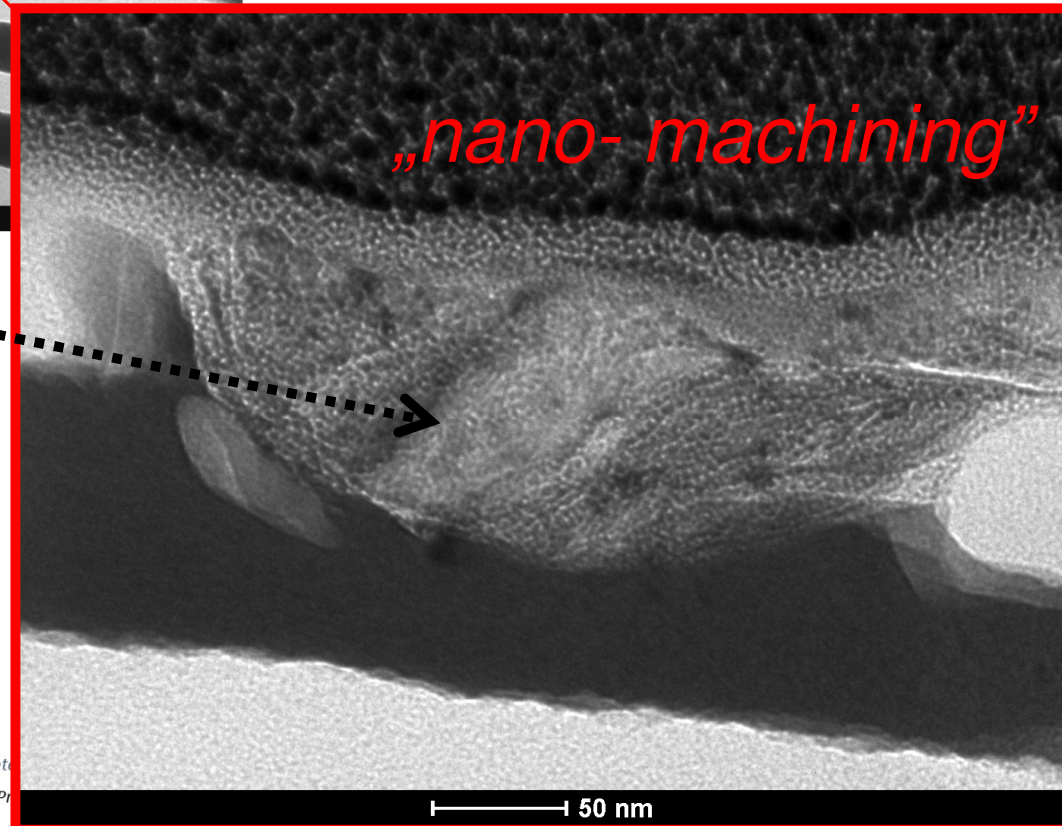
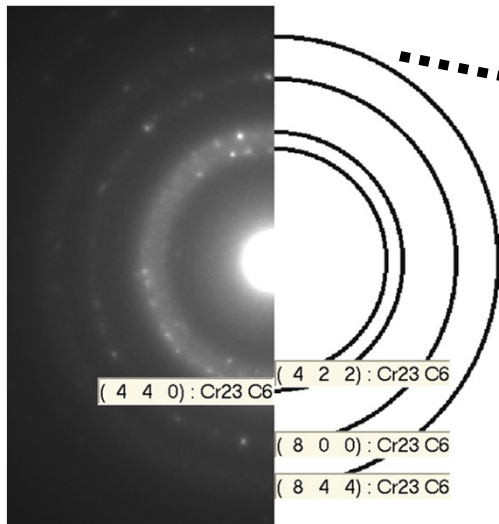
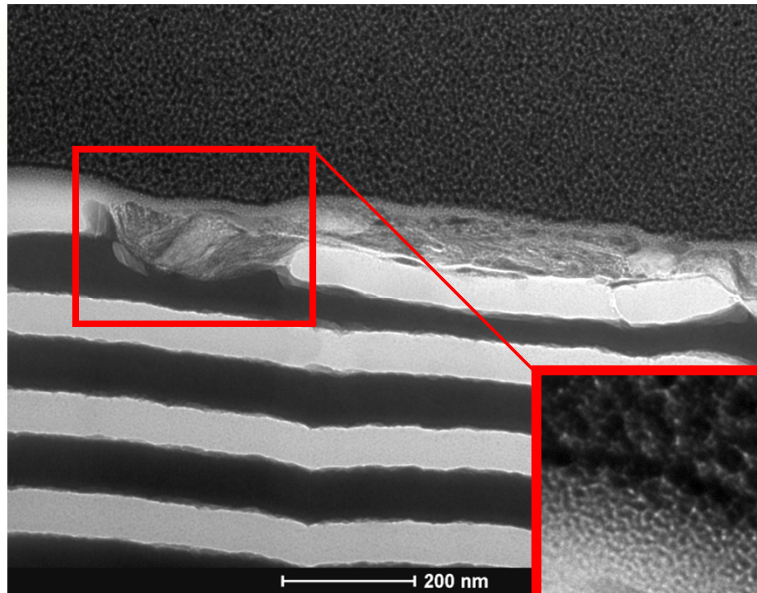


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- Project NCN nr: 3066/B/T02/2011/40- *FINISHED*
- Project NCN nr: 2012/06/M/ST8/00408- *HARMONIA- FINISHED*
- Project NCN nr: 2012/07/B/ST8/03396- *OPUS- FINISHED*
- Project NCN nr: 2014/15/B/ST8/00103- *OPUS- FINISHED*
- Project NCN nr: 2015/19/B/ST8/00942- *OPUS- in progress*
- Project NCBR, number: DZP/M-ERA.NET-2015/285/2016- *in progress*



Title: Biomimetic, self-healing, multilayer structures elaboration on thermoplastic polymer materials

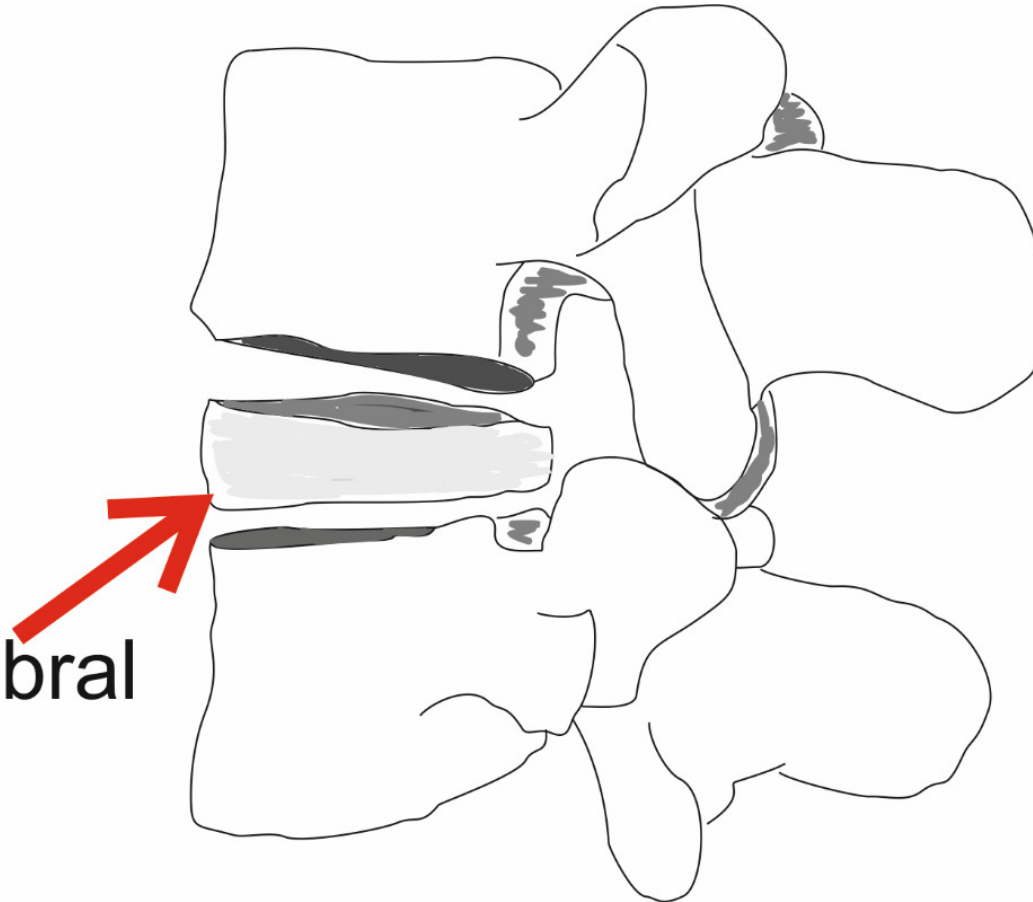
Project WND-POWR.03.02.00-00-1043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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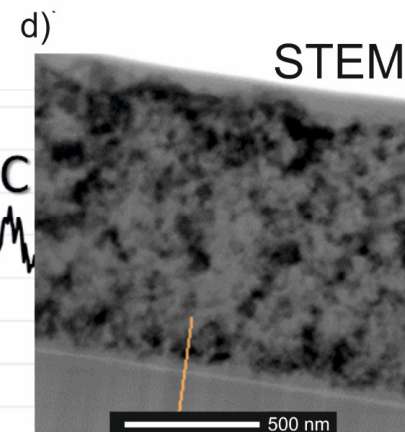
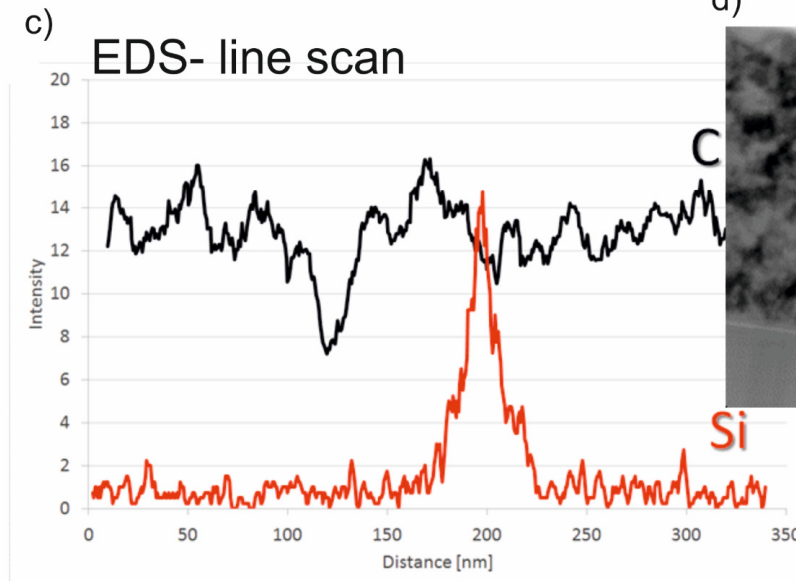
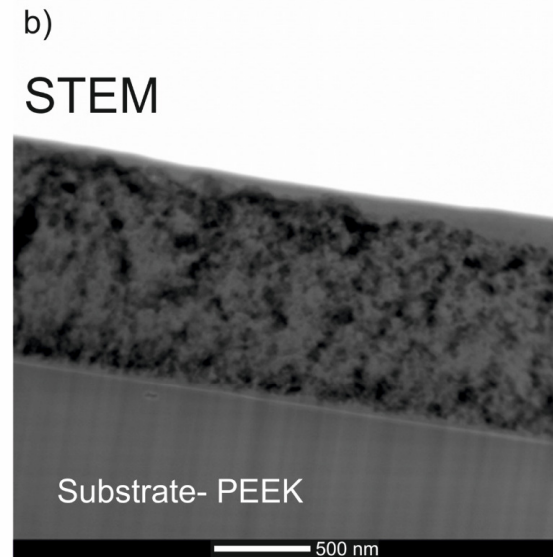
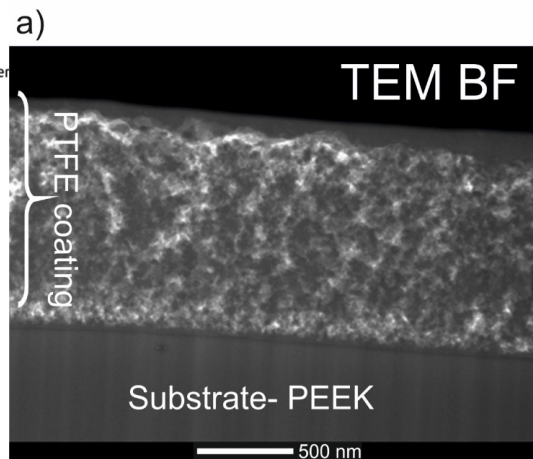
intervertebral
disc
implant



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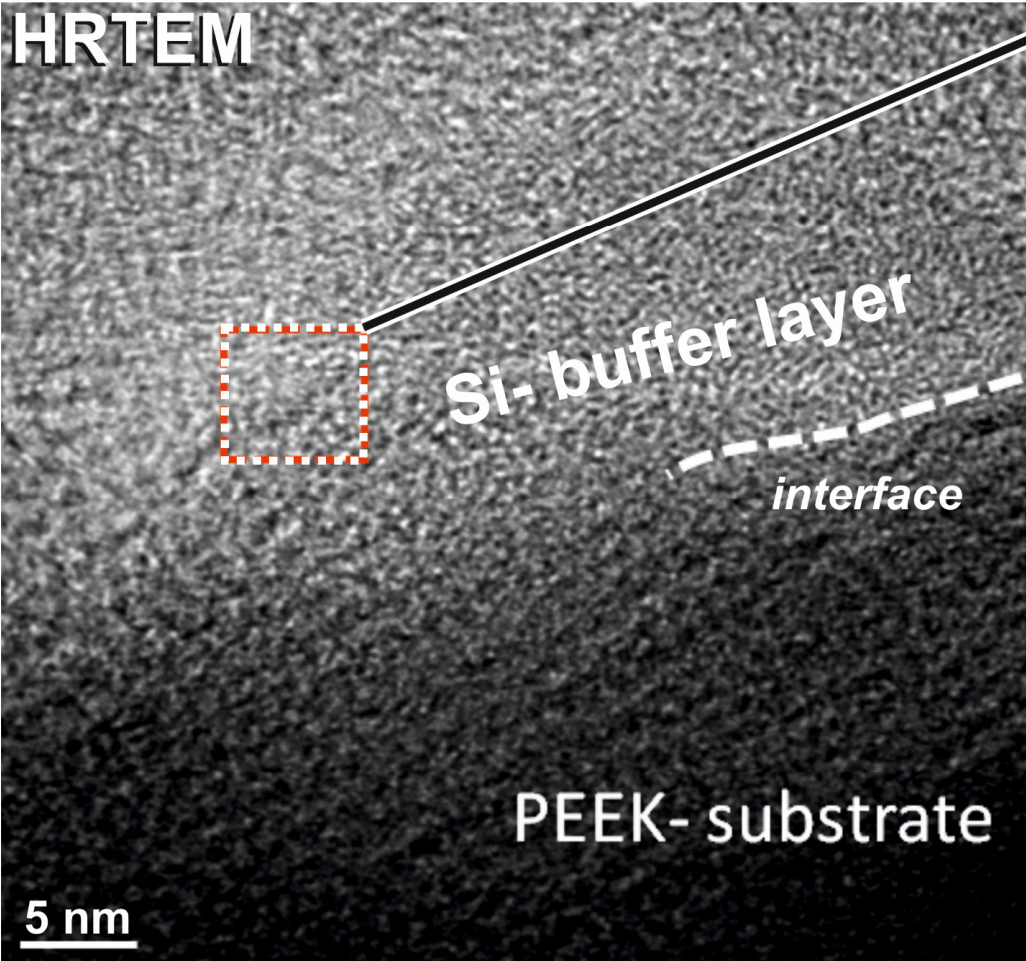
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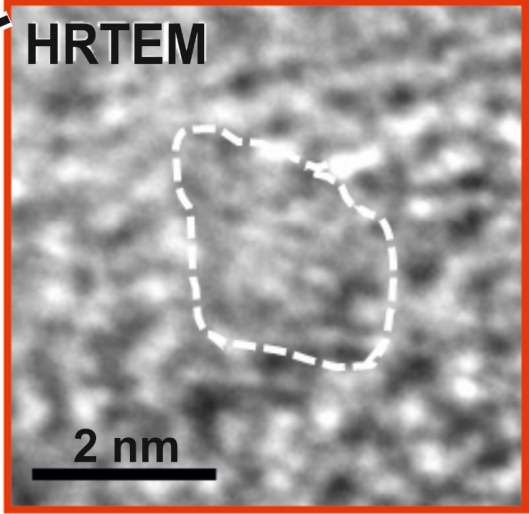
a)

HRTEM



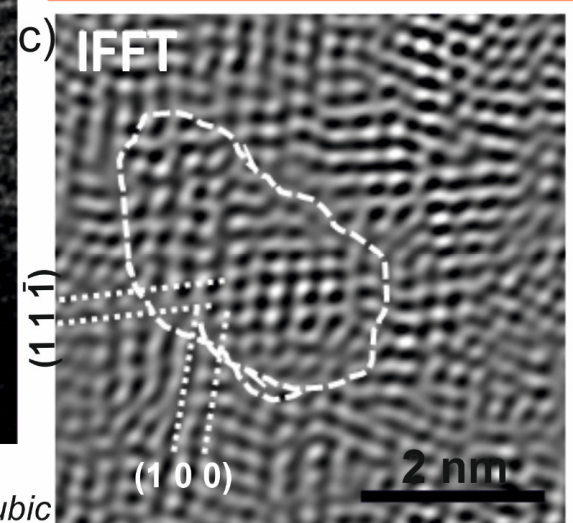
b)

HRTEM



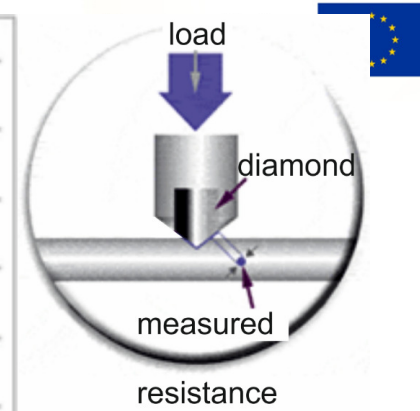
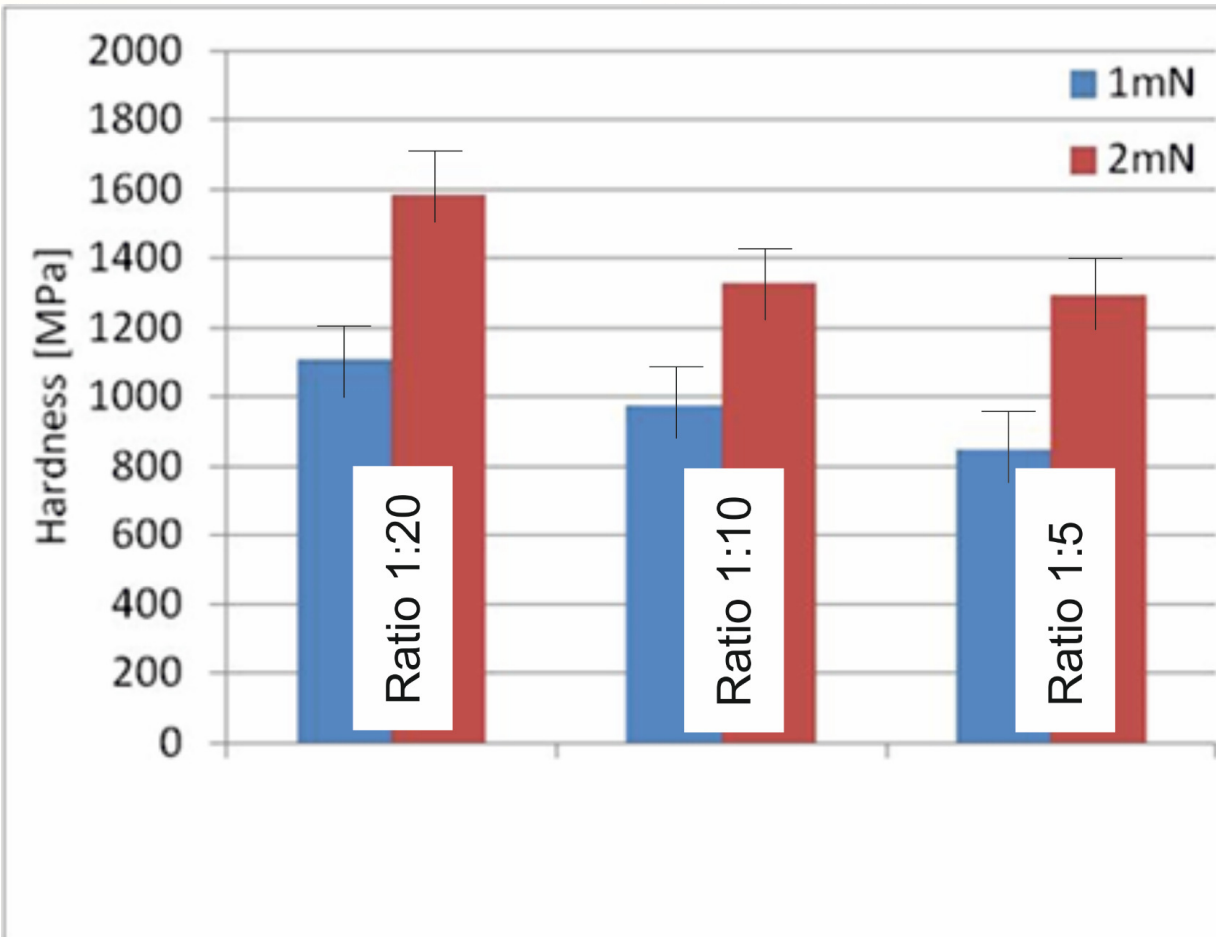
c)

IFFT

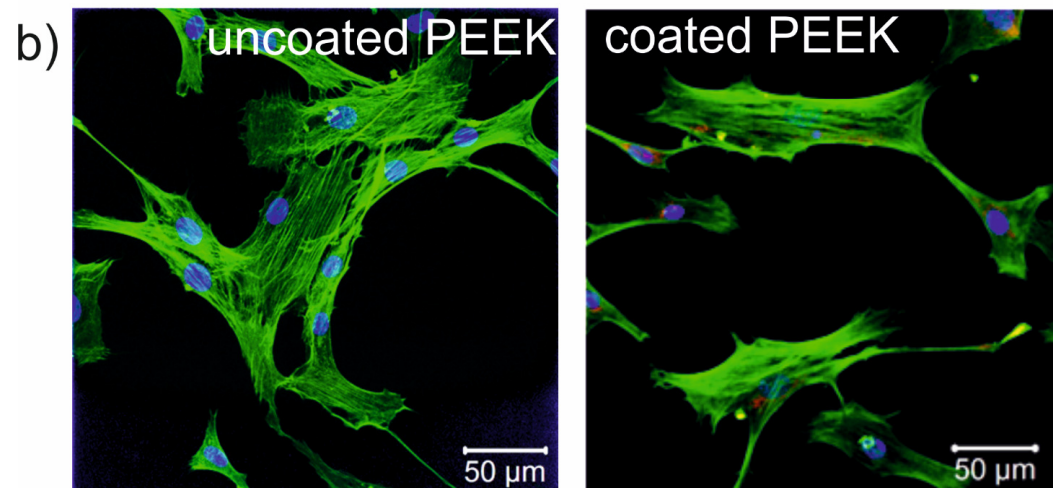
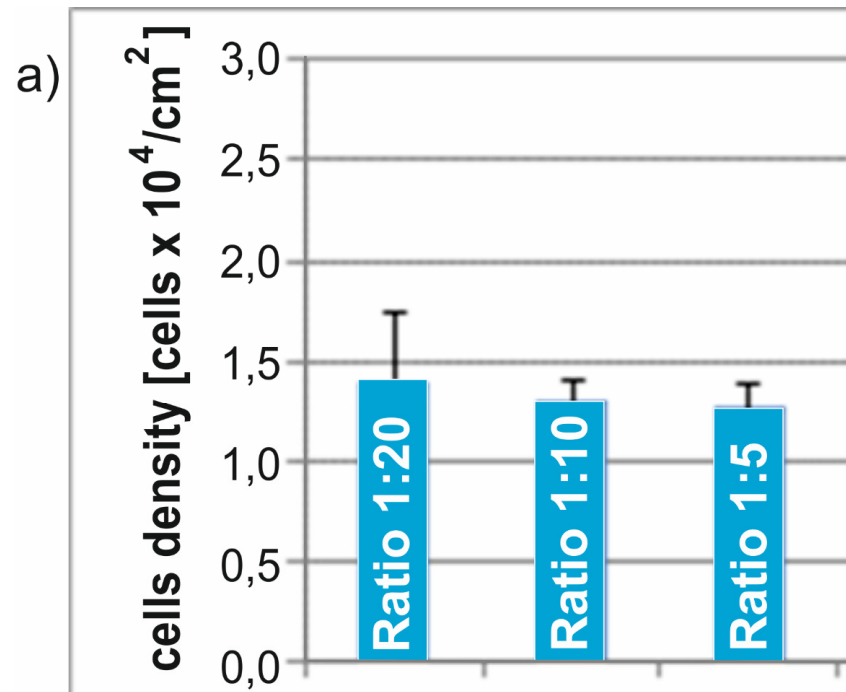


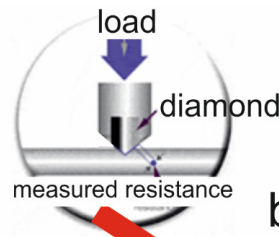
Zone axis
[0 1 1] Si- cubic

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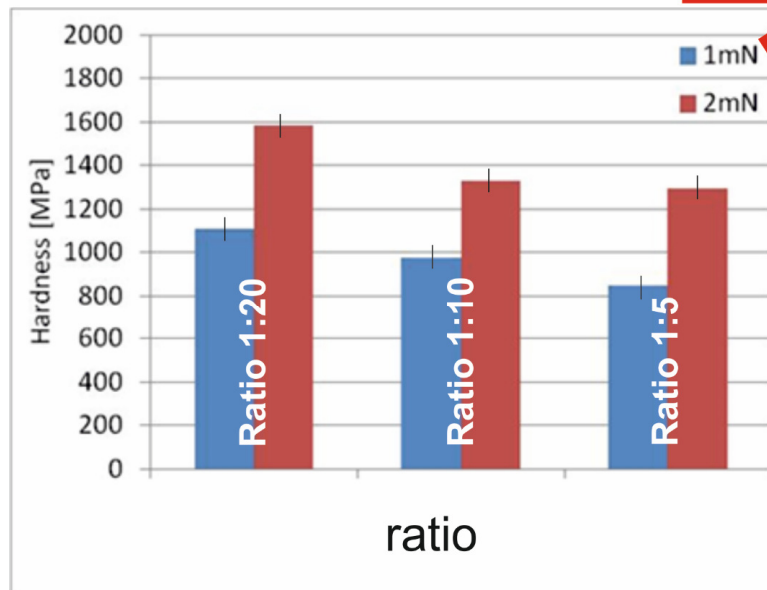


Different ratio

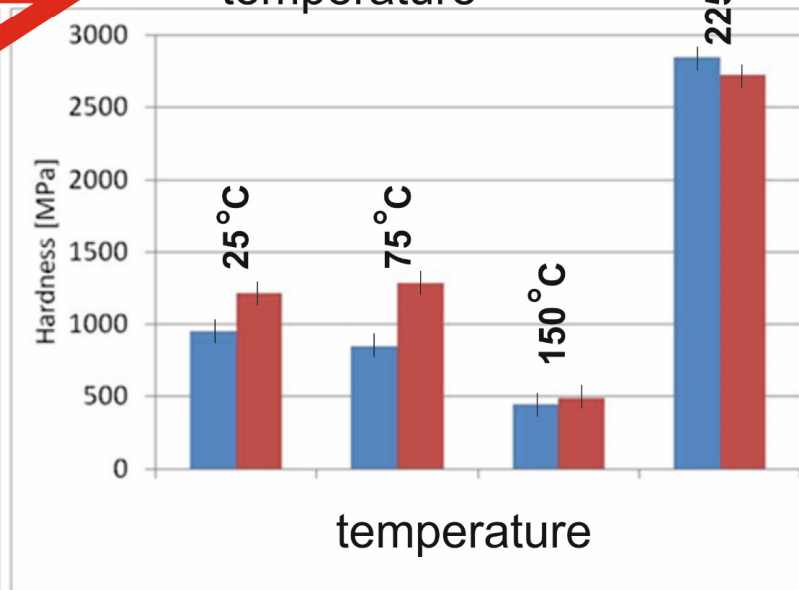





a) different phases ratio



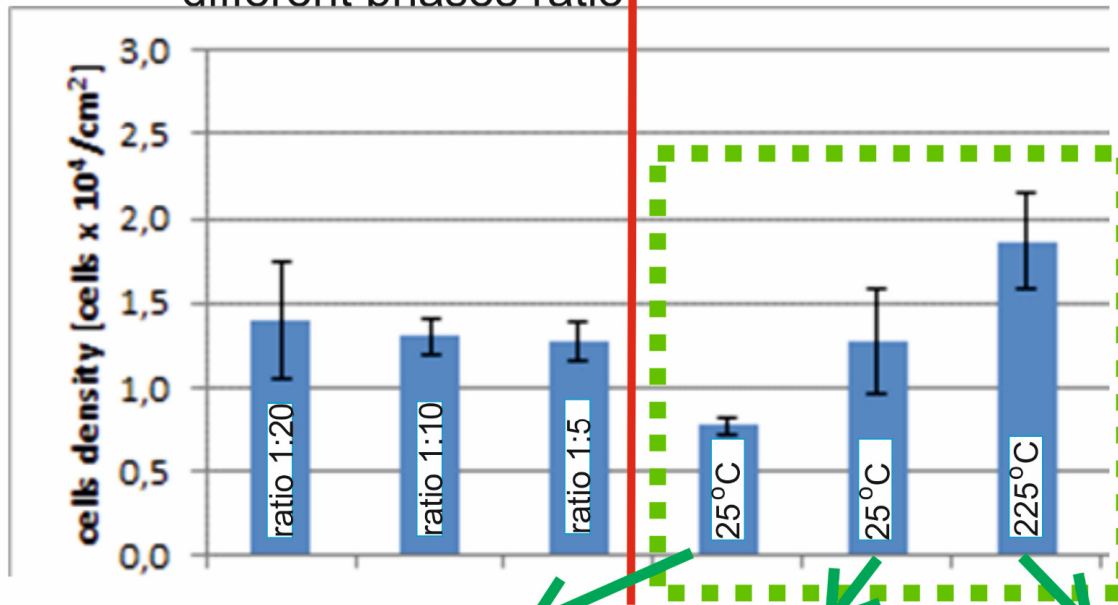
b) different deposition temperature



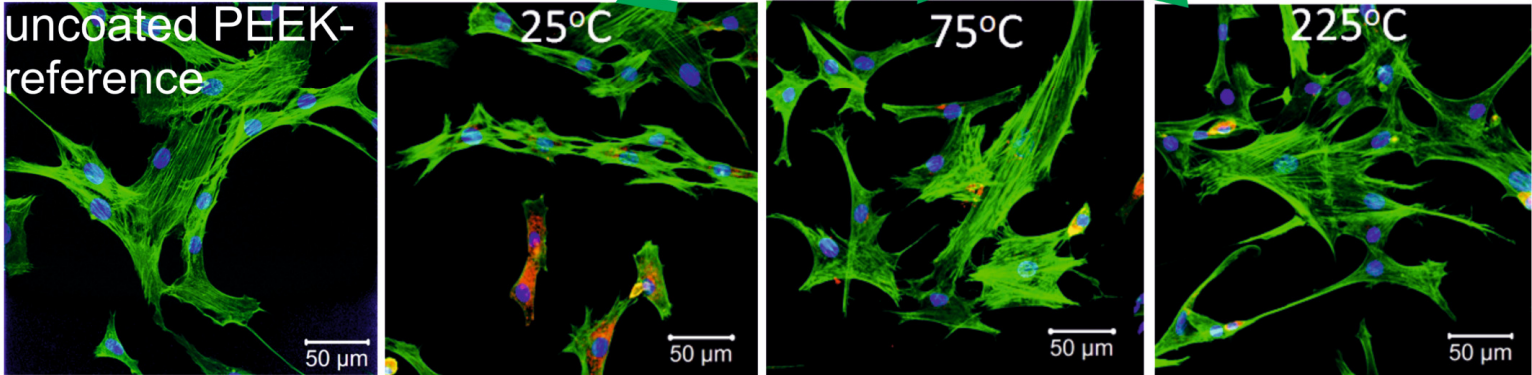


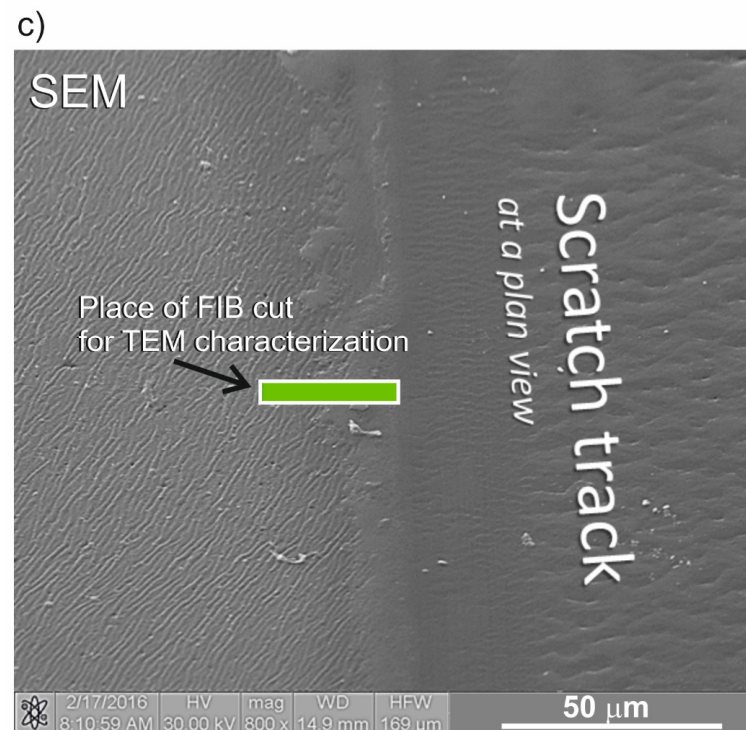
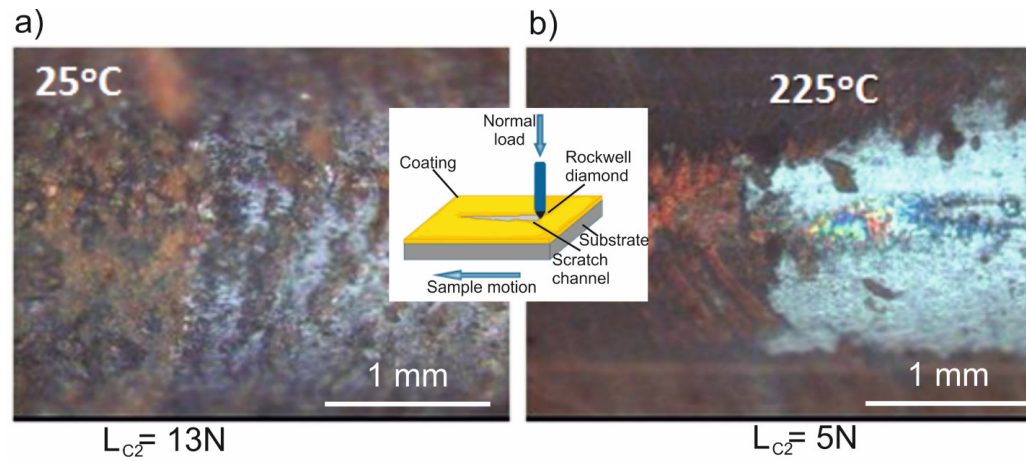
a)  b) different deposition temperature

different phases ratio

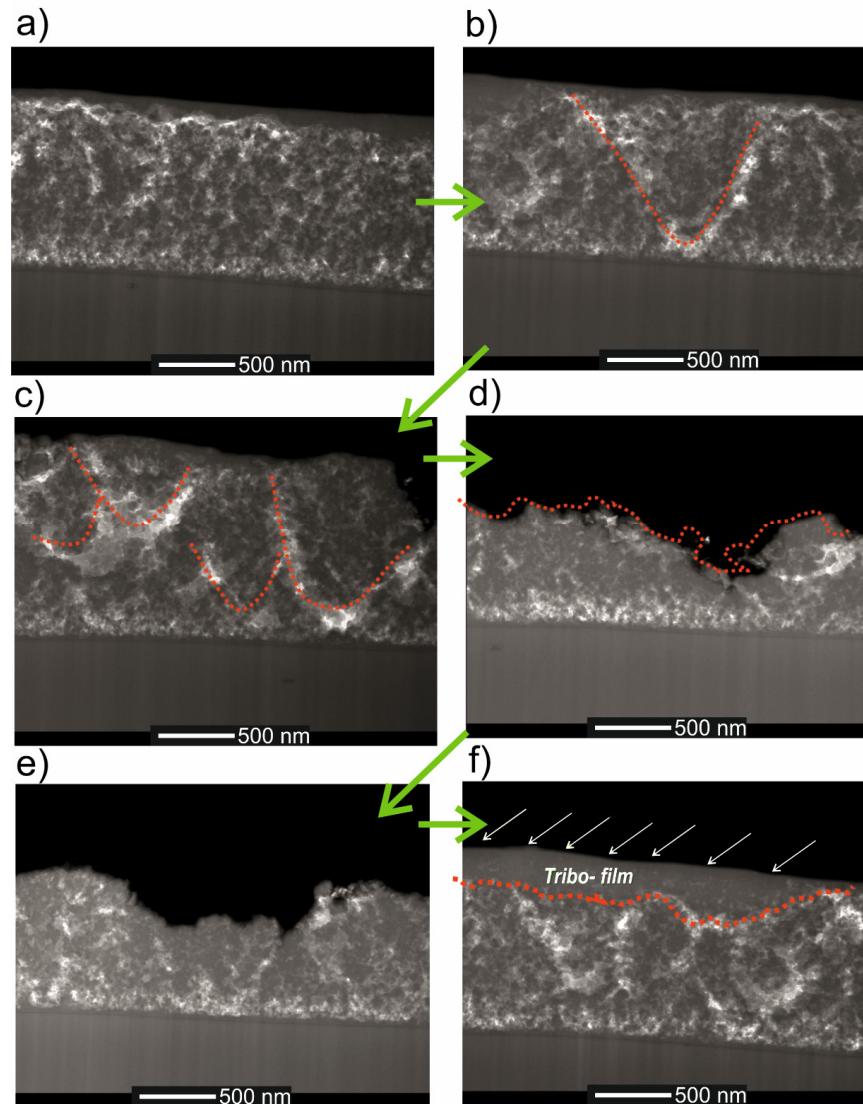


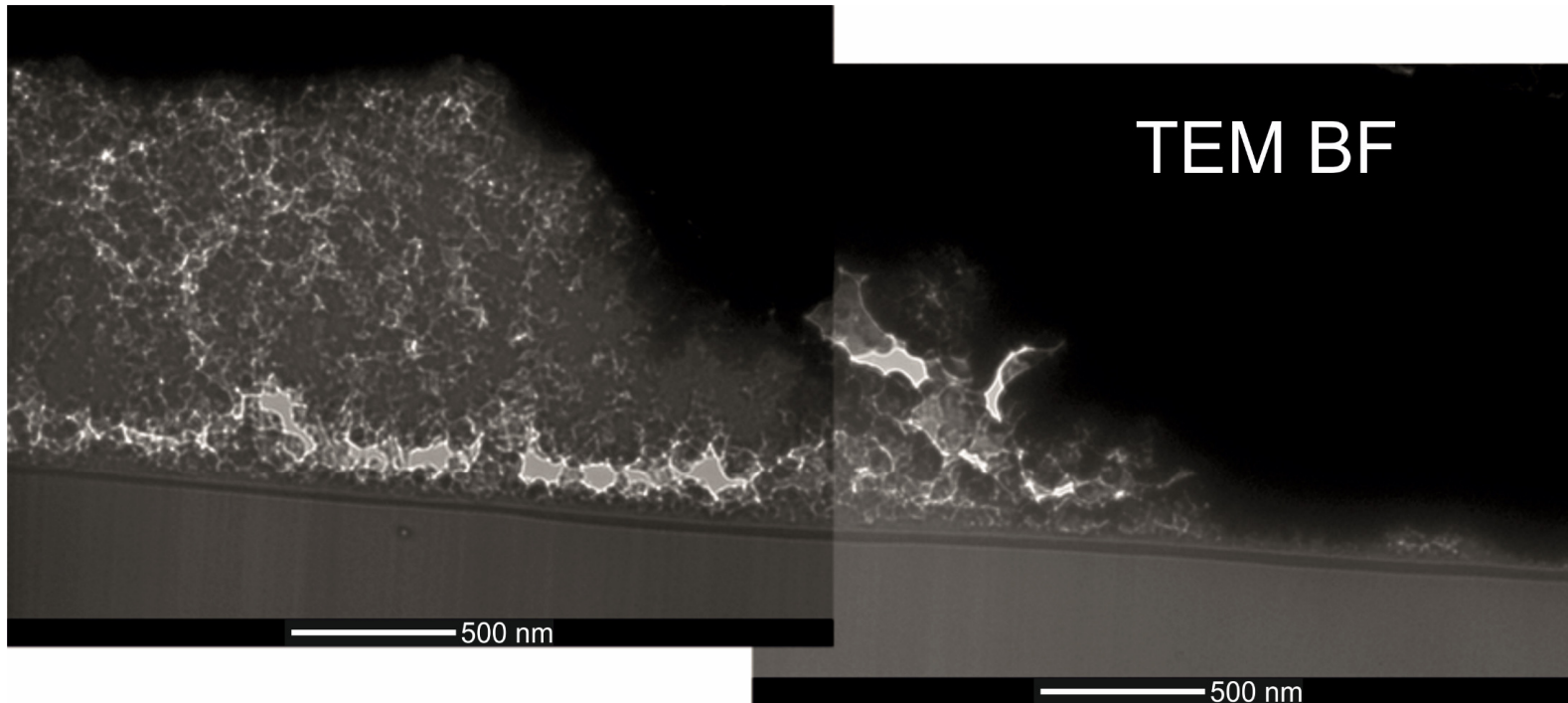
c) uncoated PEEK-reference





TEM BF

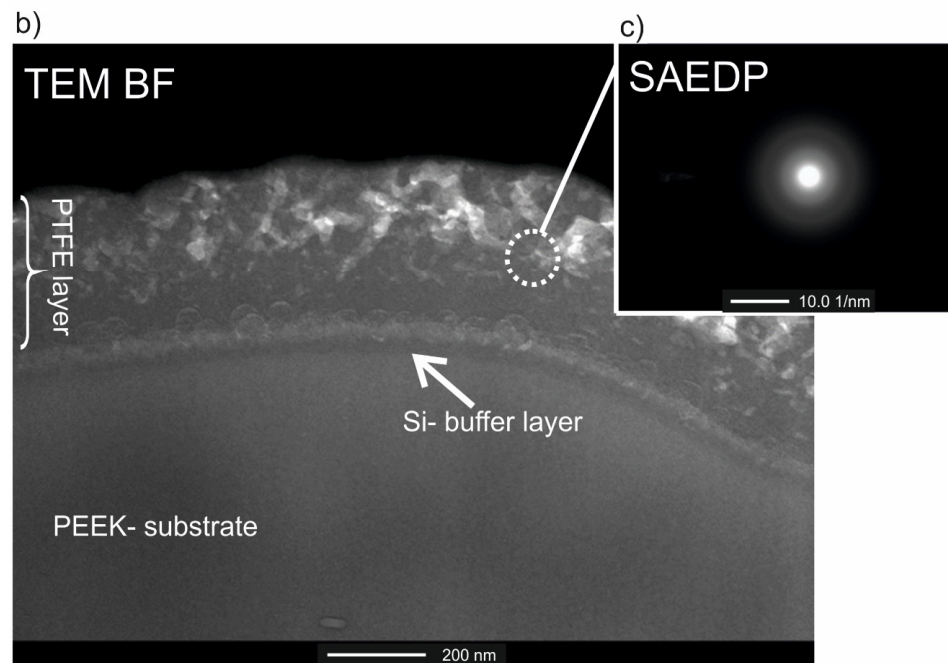
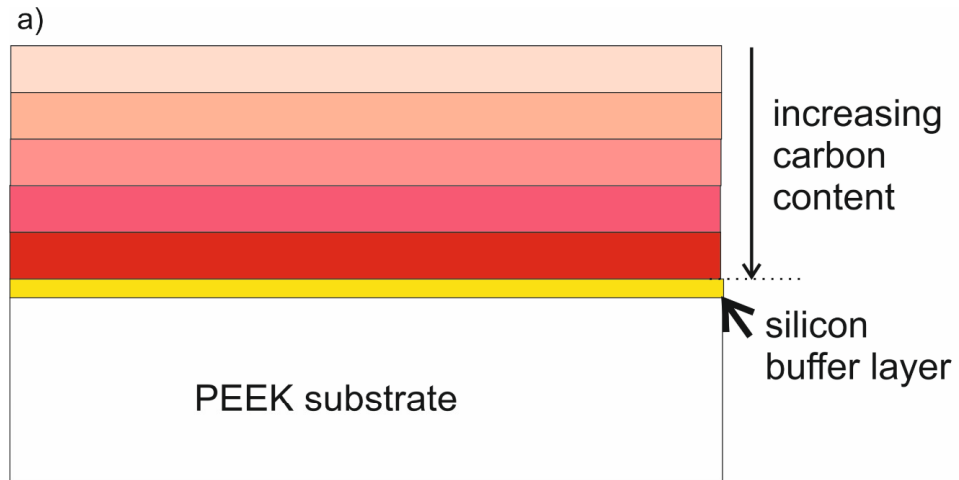


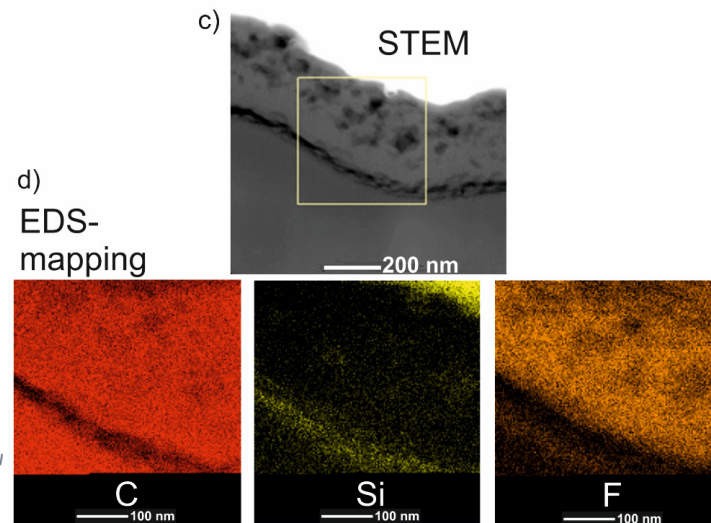
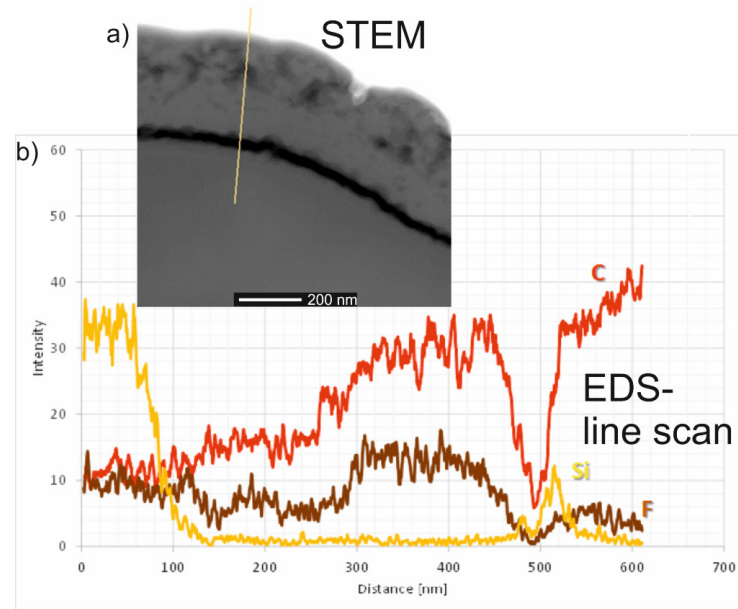


Project WND-POWR.03.02.00-00-1043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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porous structure-
responsible
for cell proliferation

Bio-zone

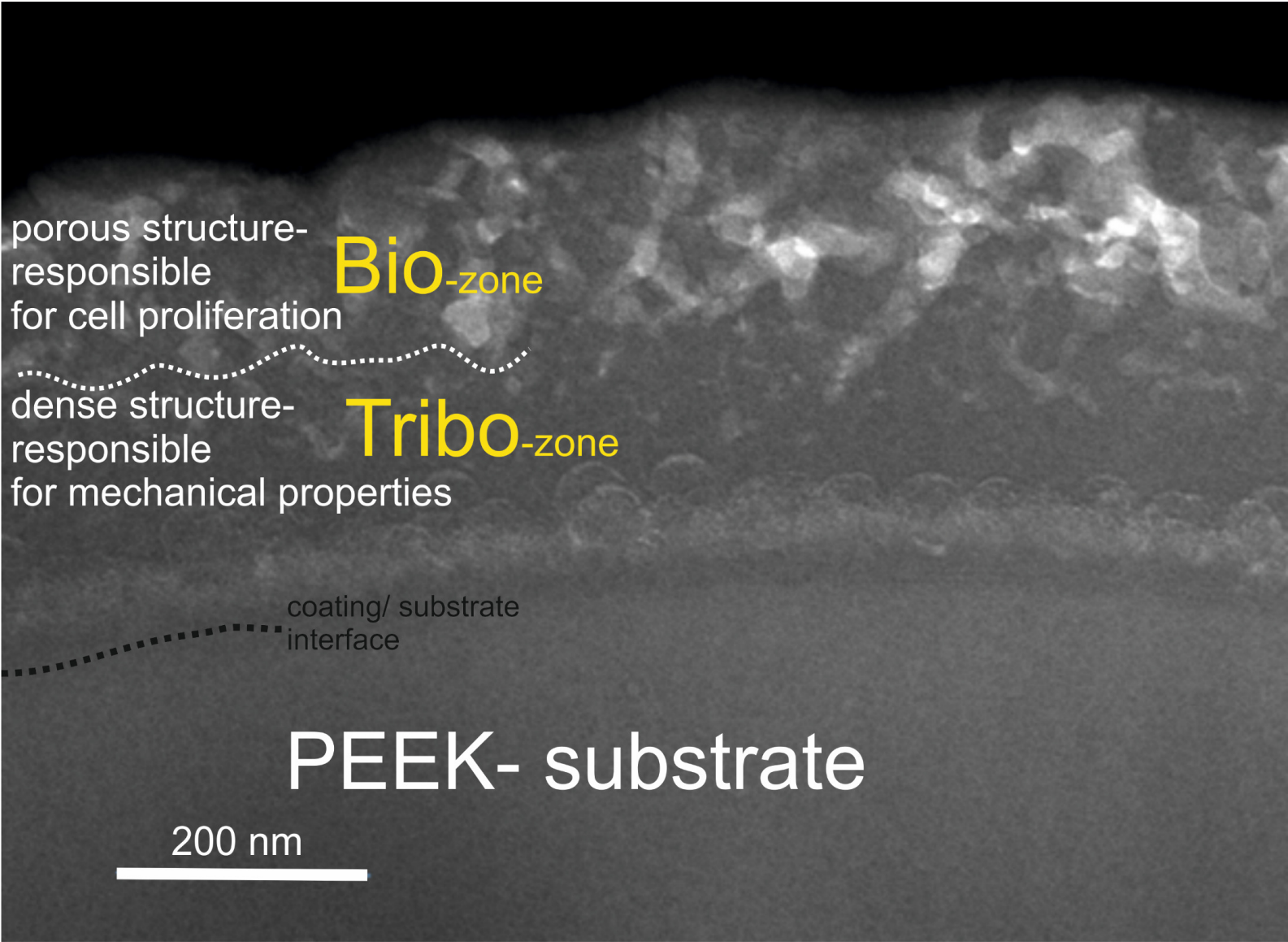
dense structure-
responsible
for mechanical properties

Tribo-zone

coating/ substrate
interface

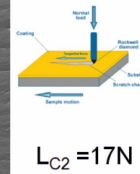
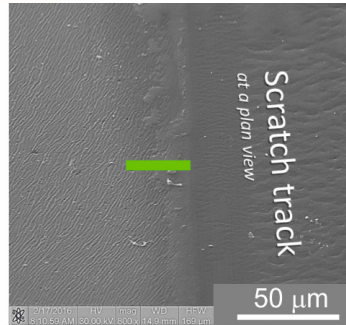
PEEK- substrate

200 nm



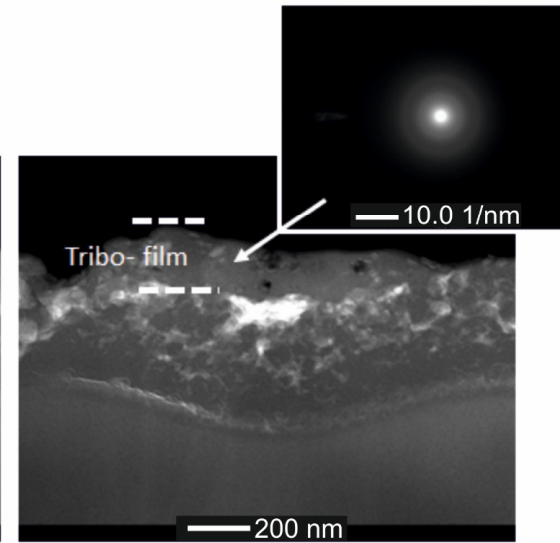
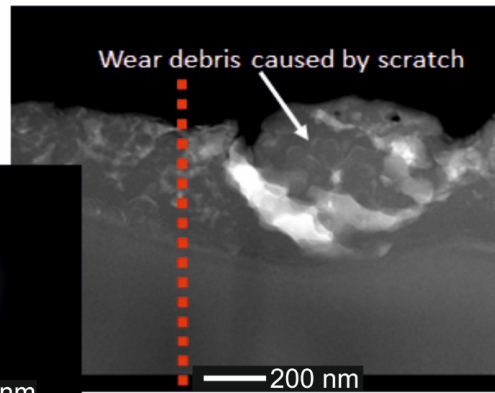
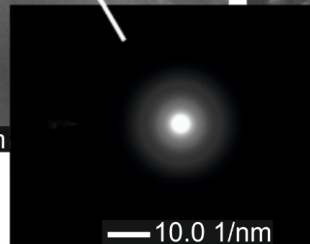
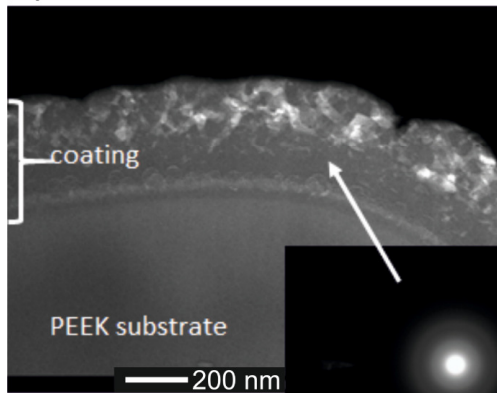


a) SEM



Scratch track-
plan view

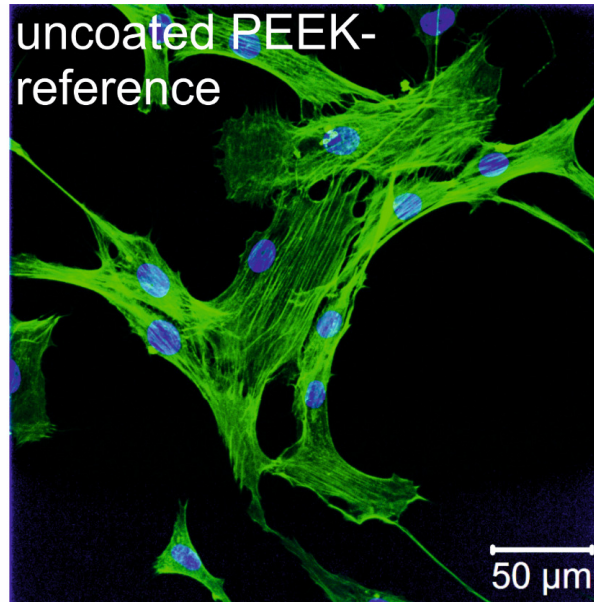
b) TEM BF



Scratch track
at the cross section

$L_{C2} = 17N$

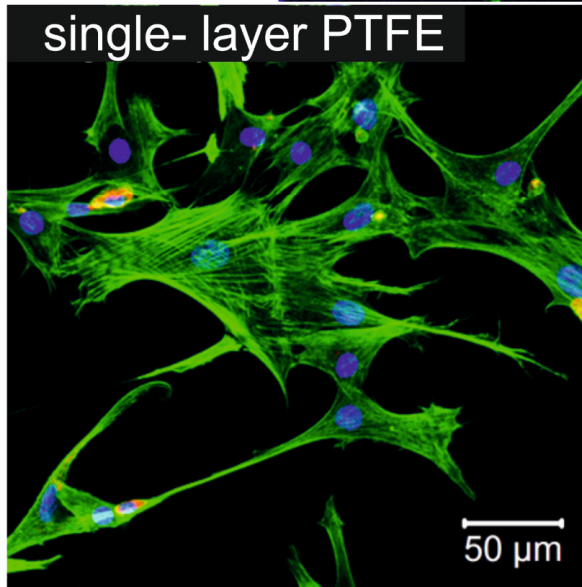
a) uncoated PEEK-
reference



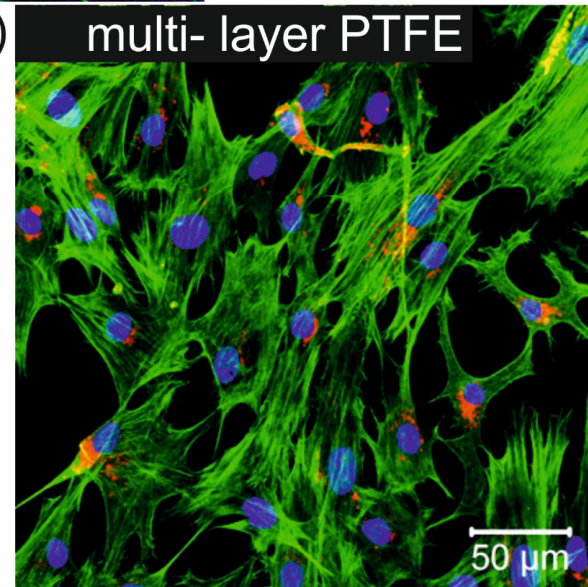
CLSM

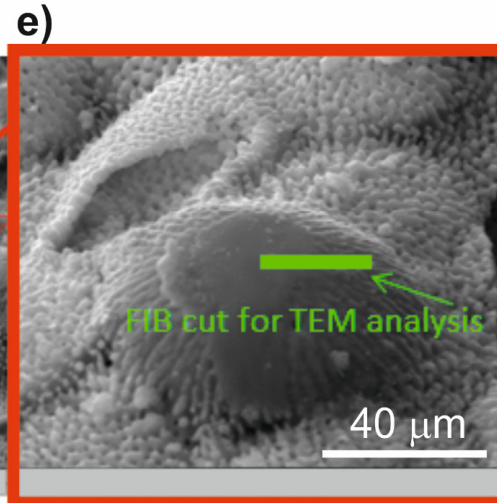
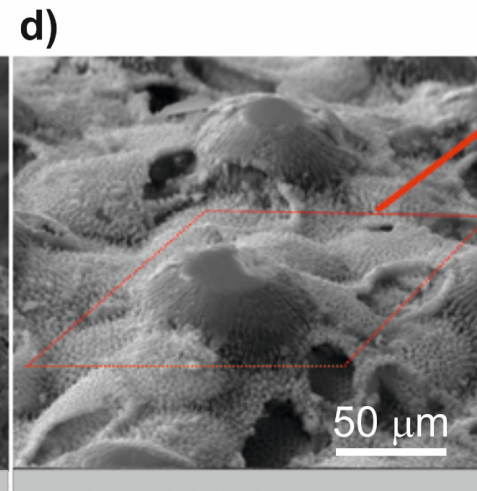
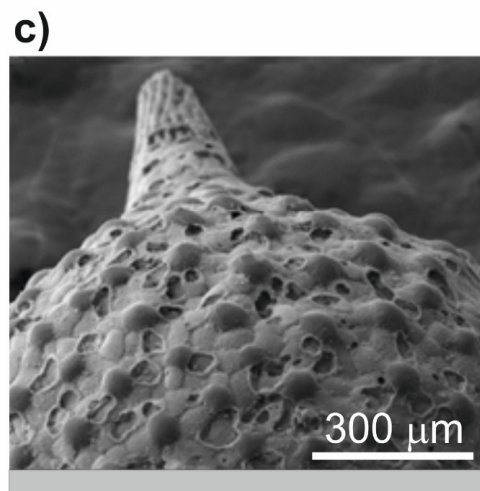
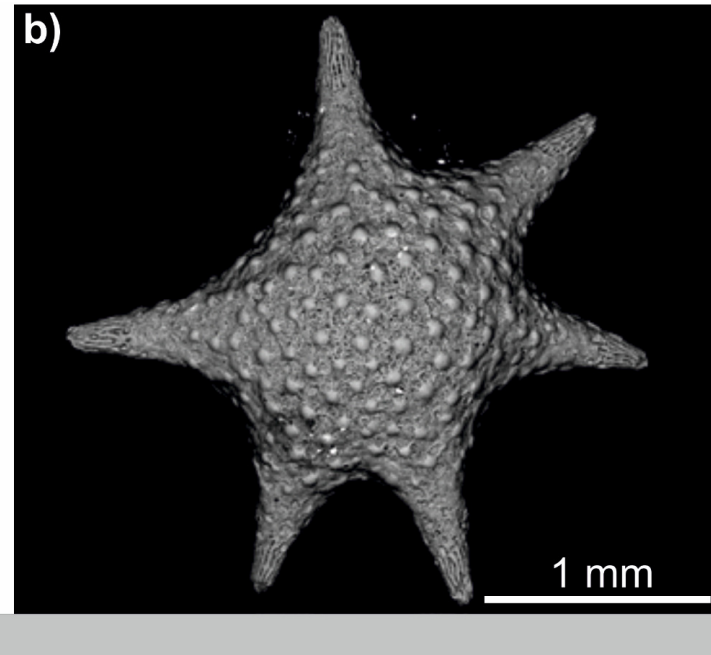
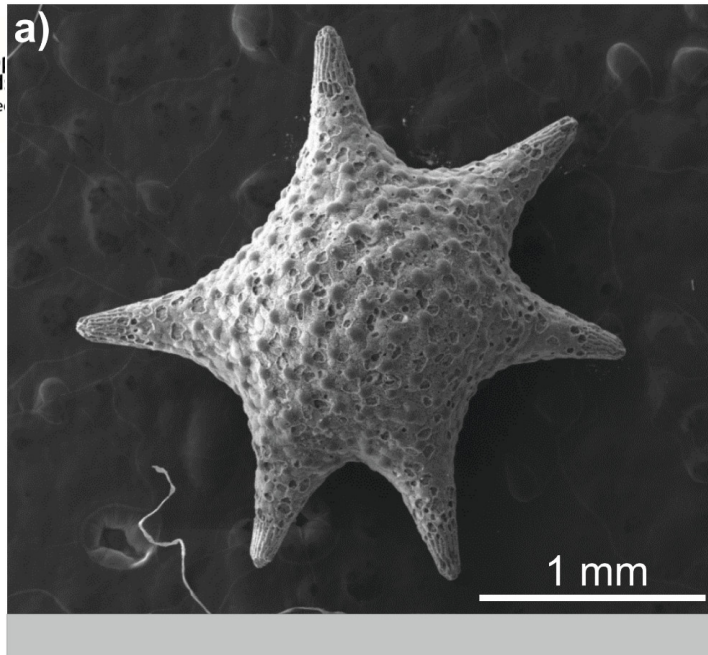


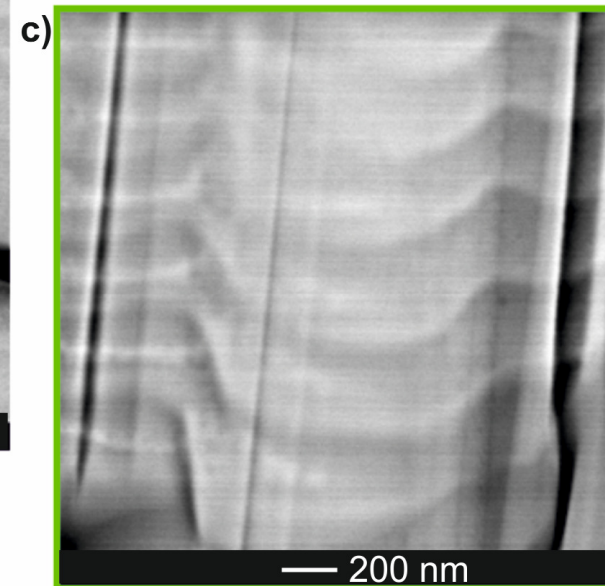
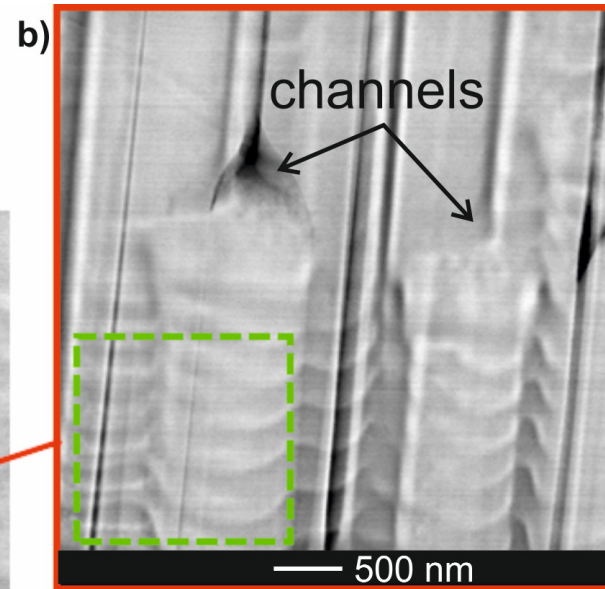
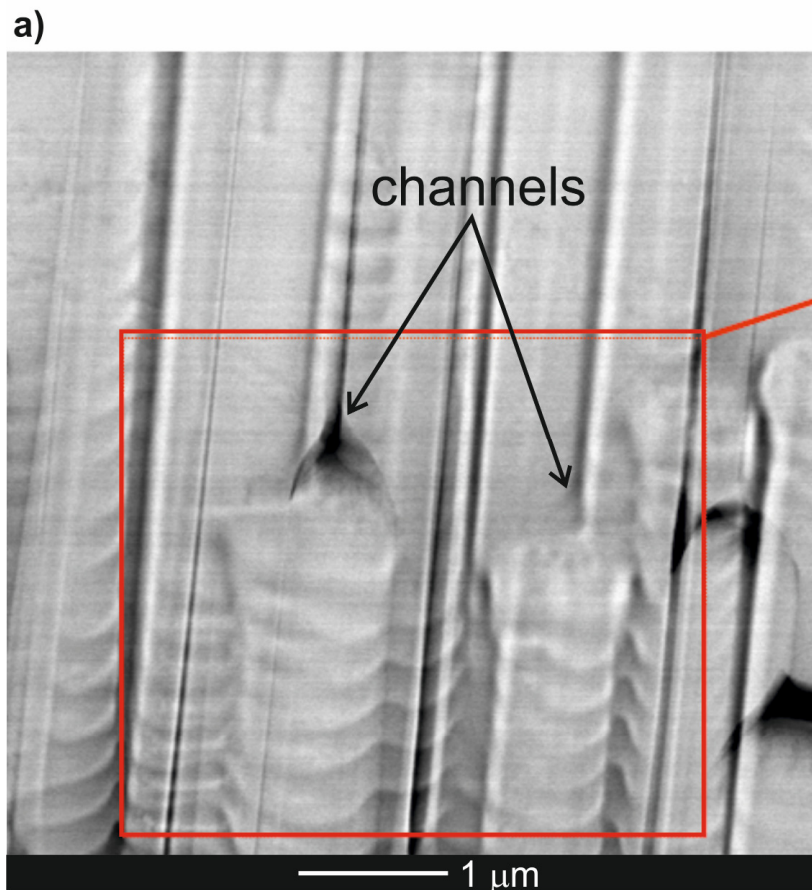
b) single- layer PTFE



c) multi- layer PTFE







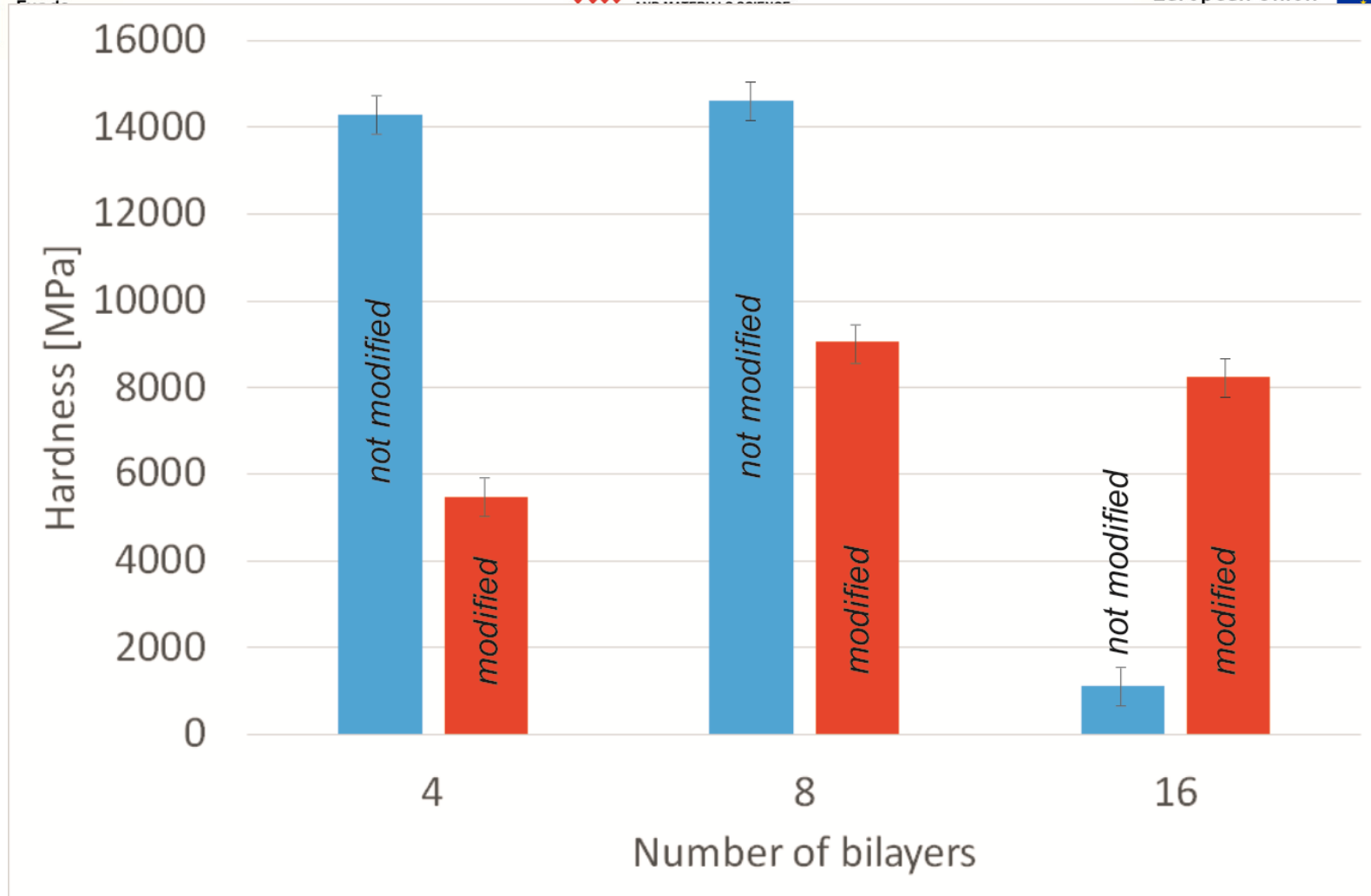


European



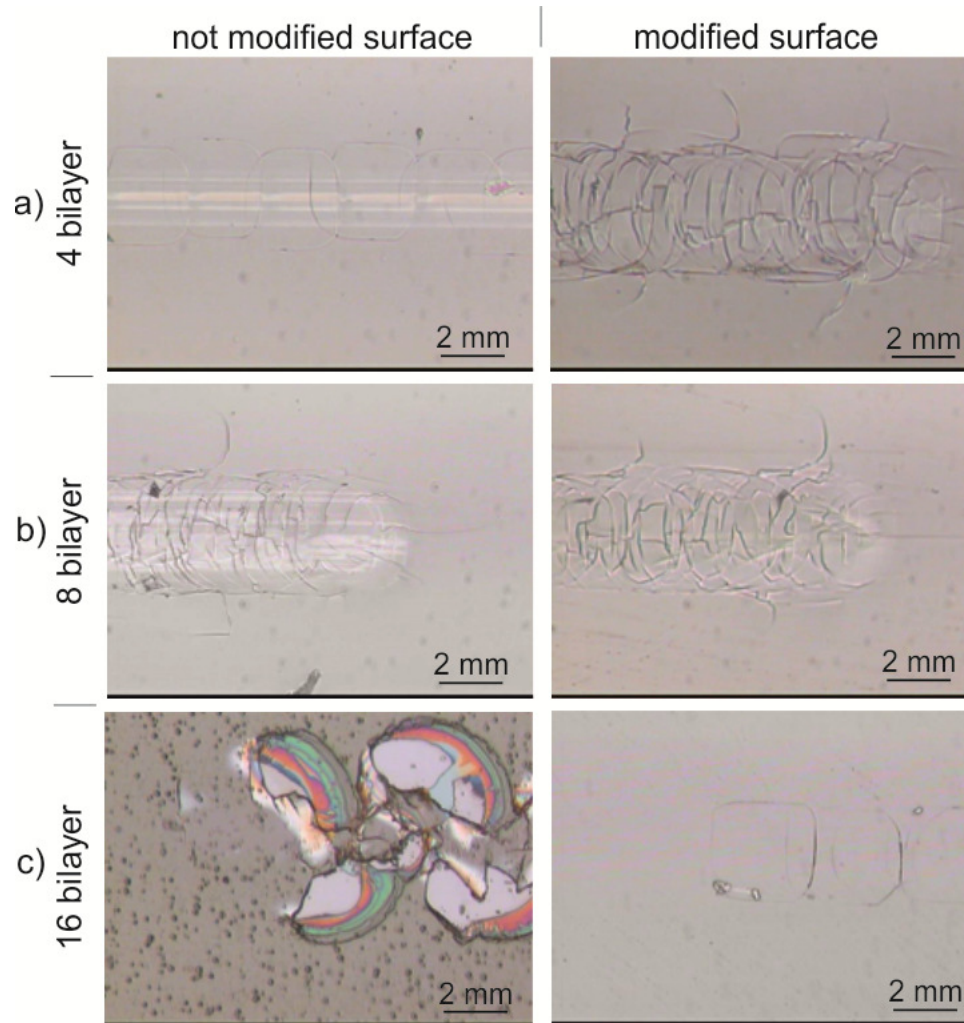
INSTITUTE OF METALLURGY

European Union

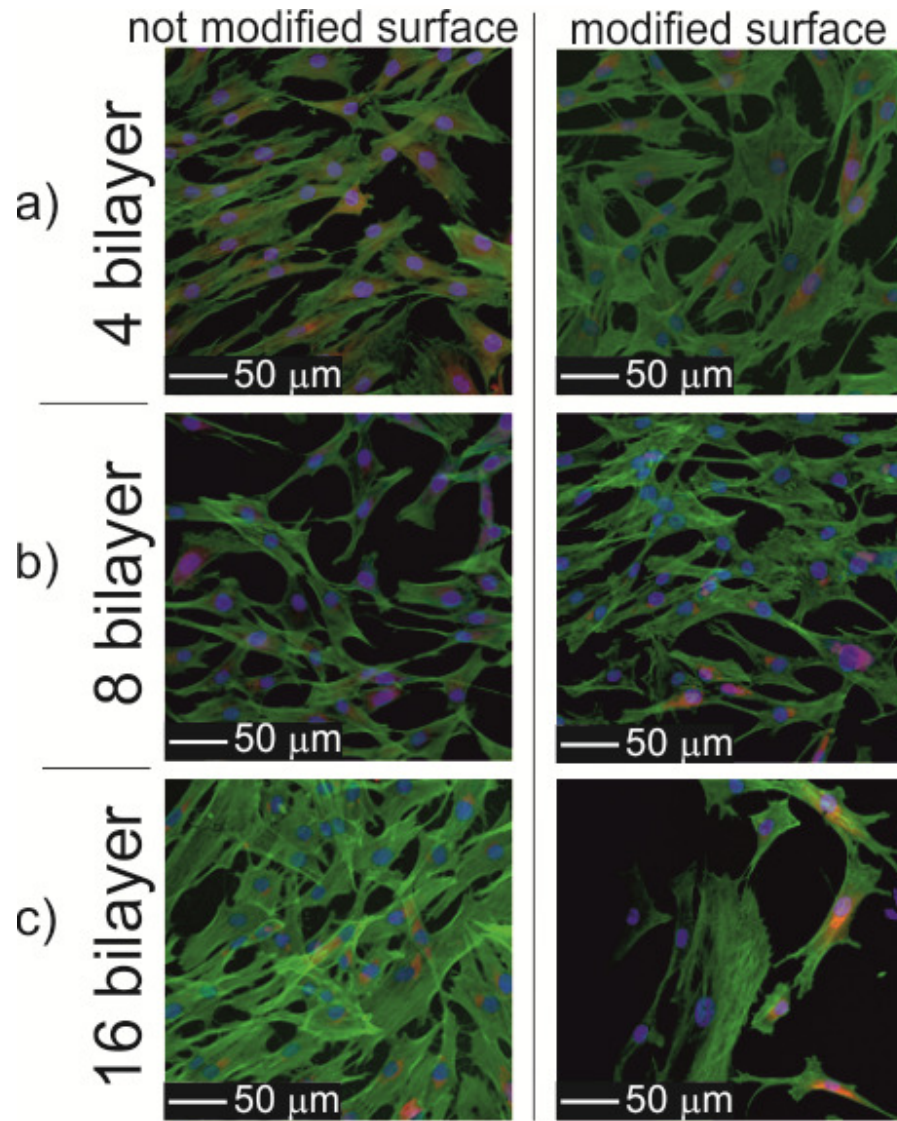


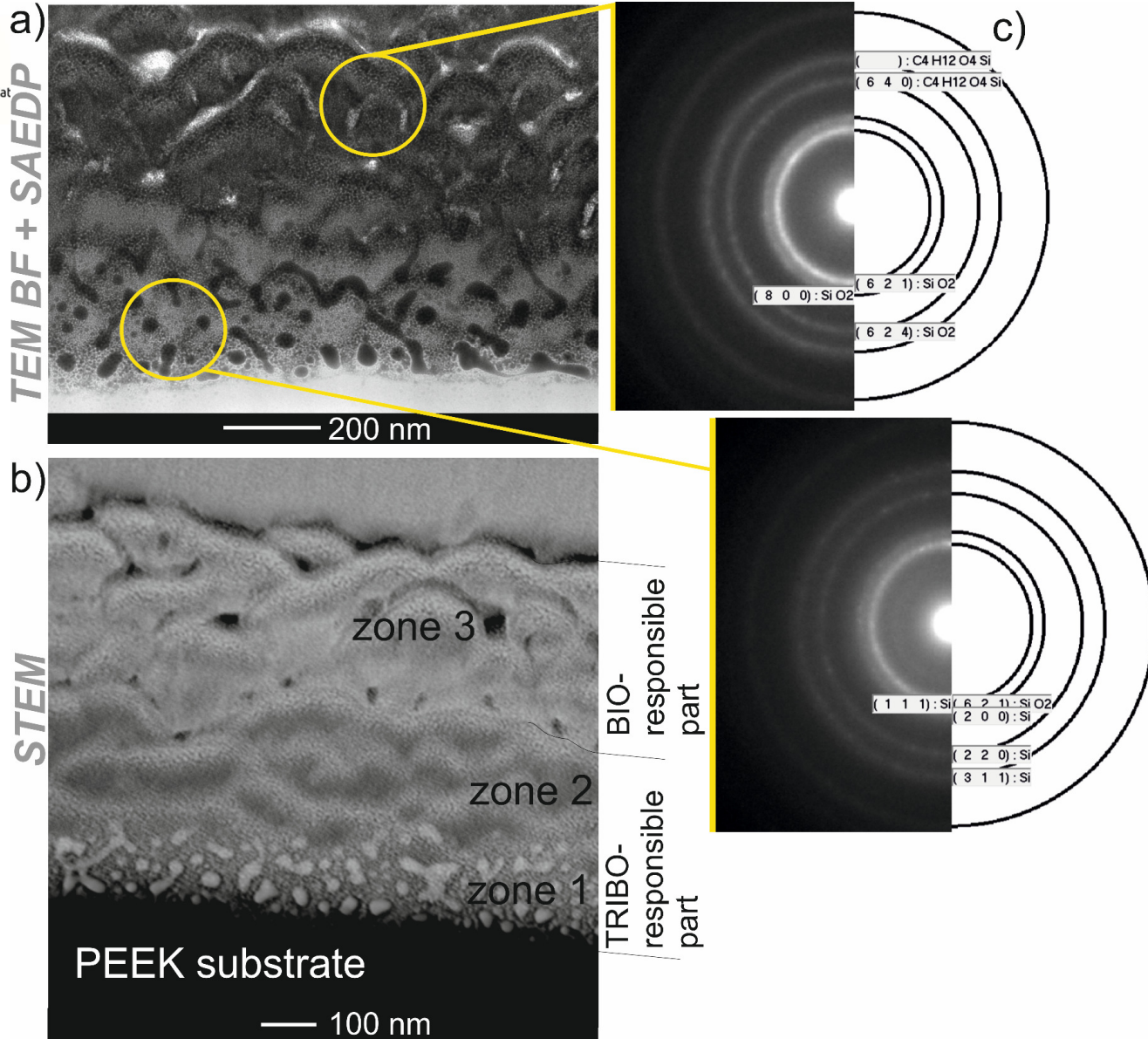
International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

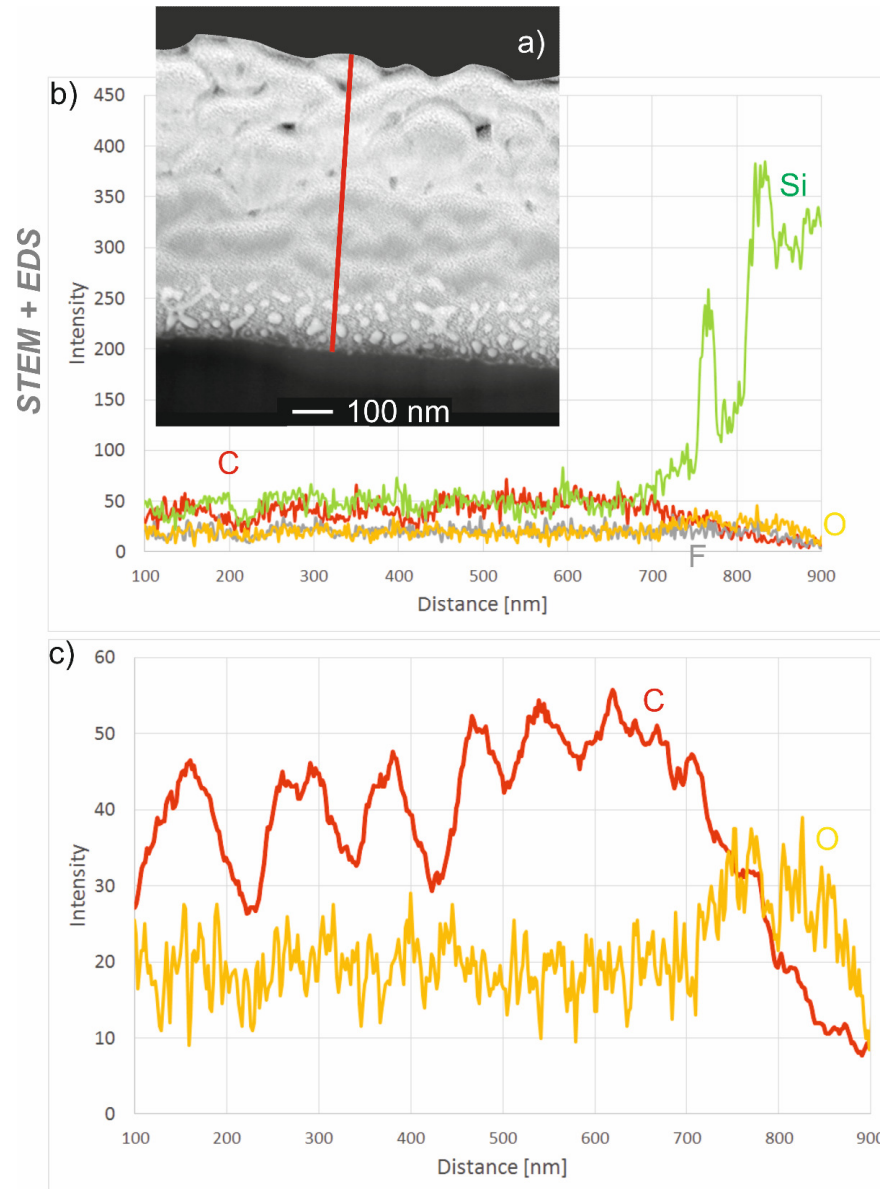
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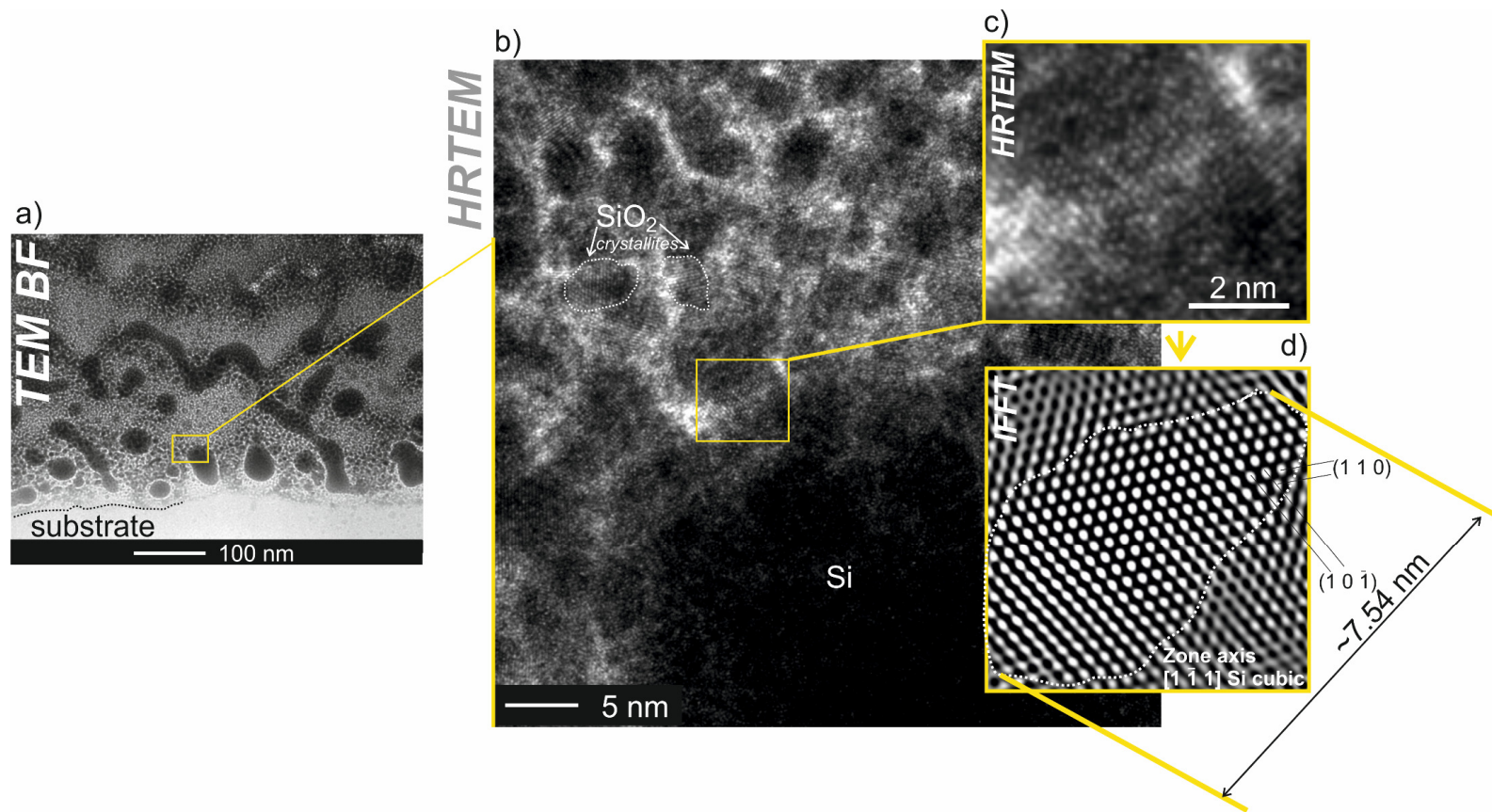


Direction of the load increase





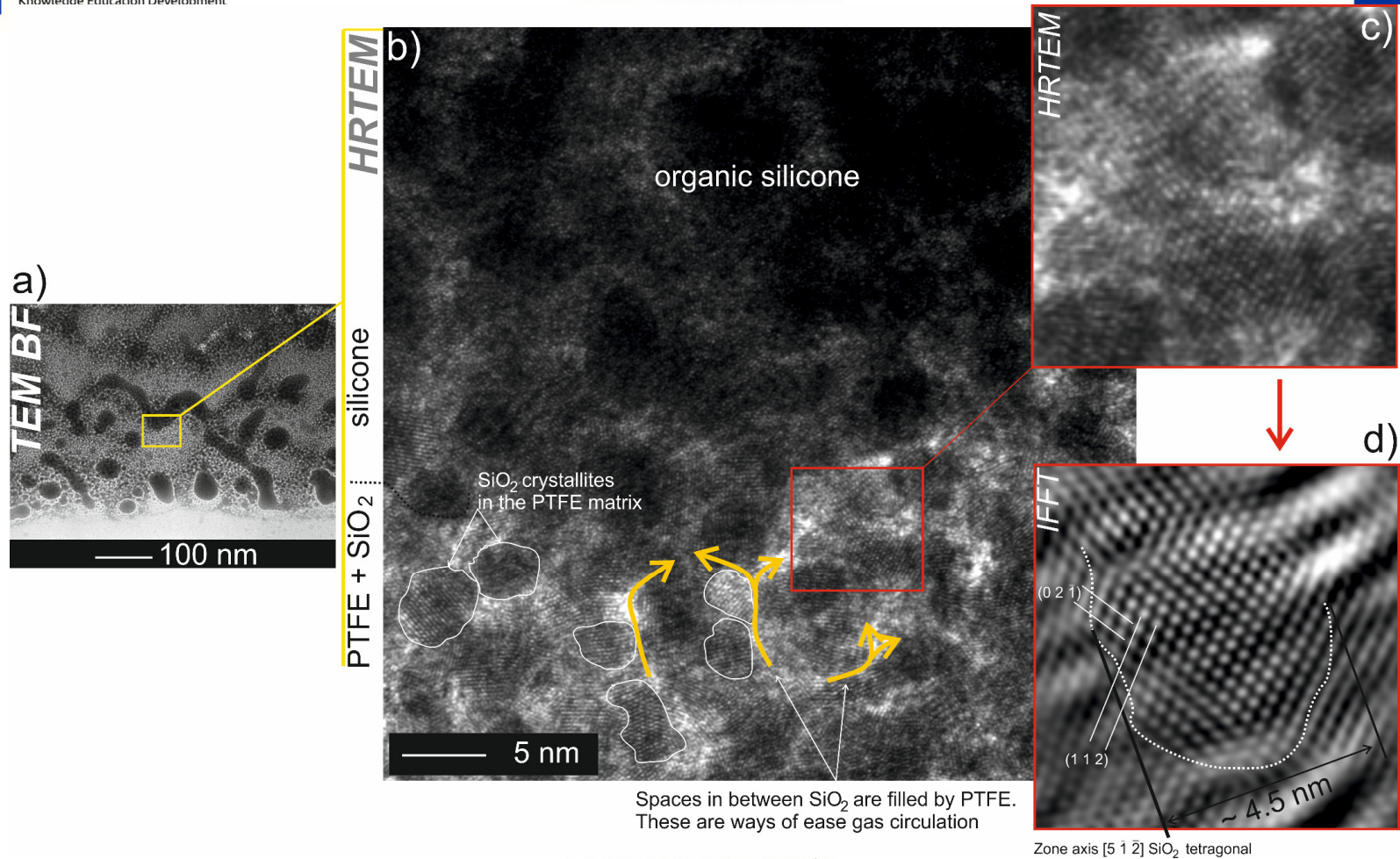




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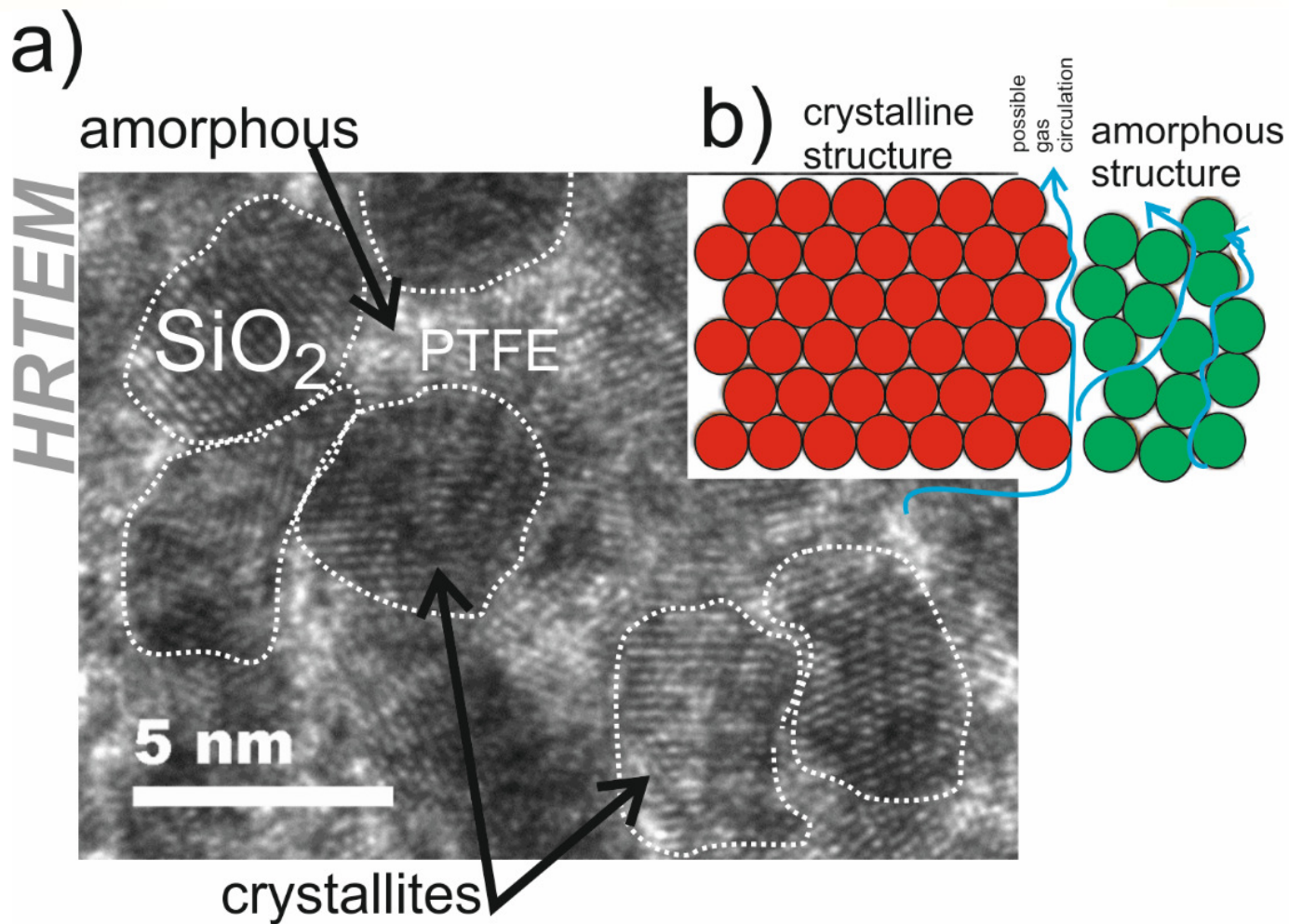
Project co-financed by the European Union within the European Social Funds

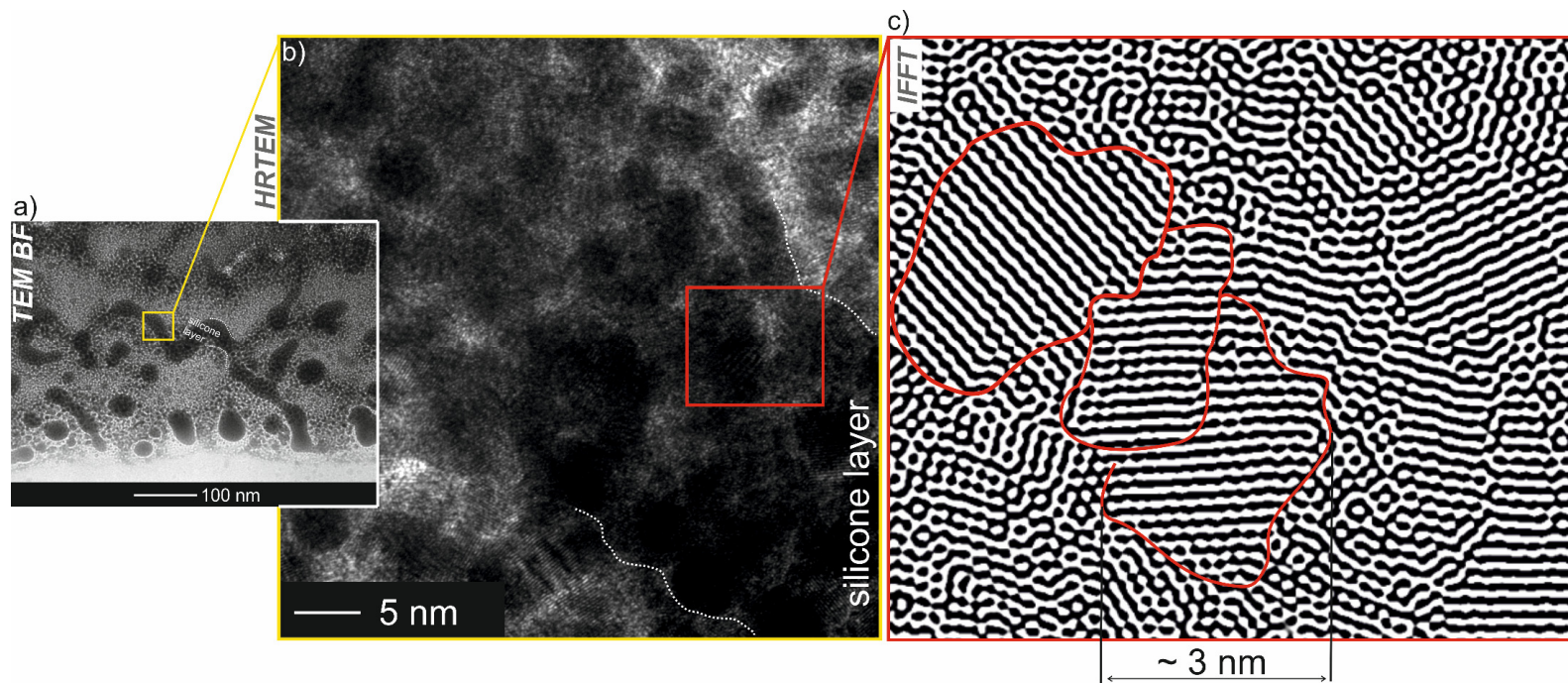


Project WND-POWR.03.02.00-00-1043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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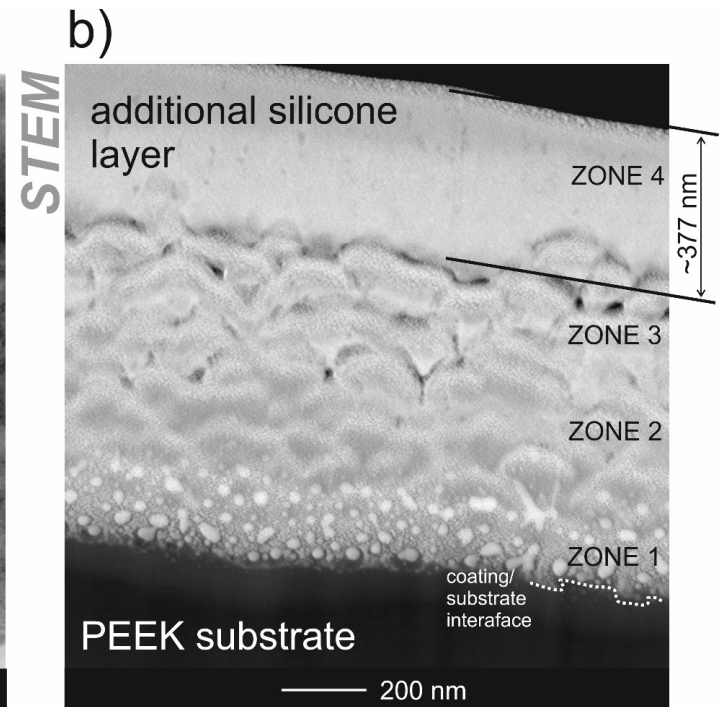
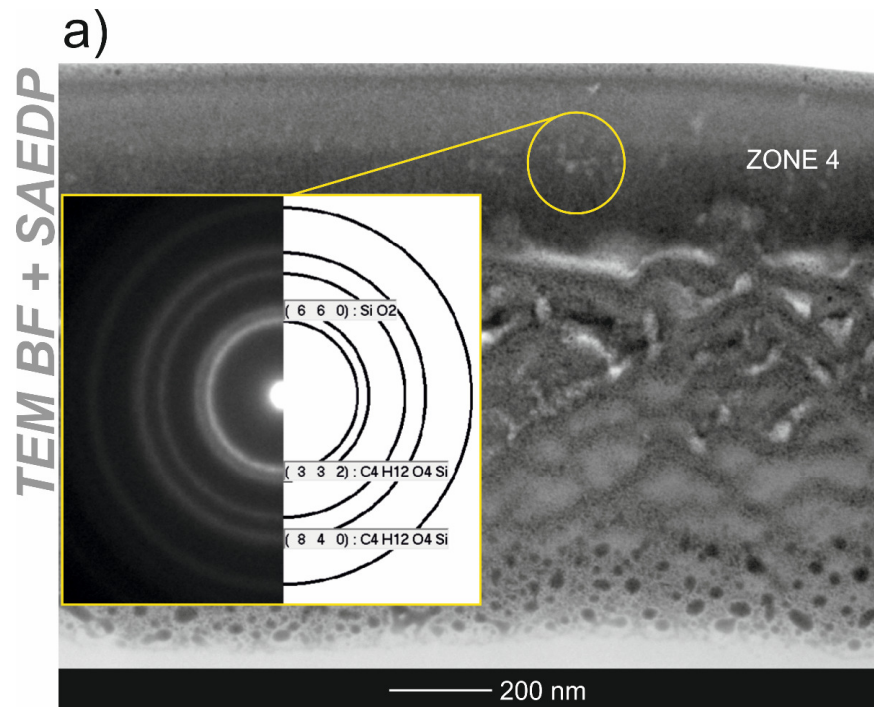




Project WND-POWR.03.02.00-00-1043/16

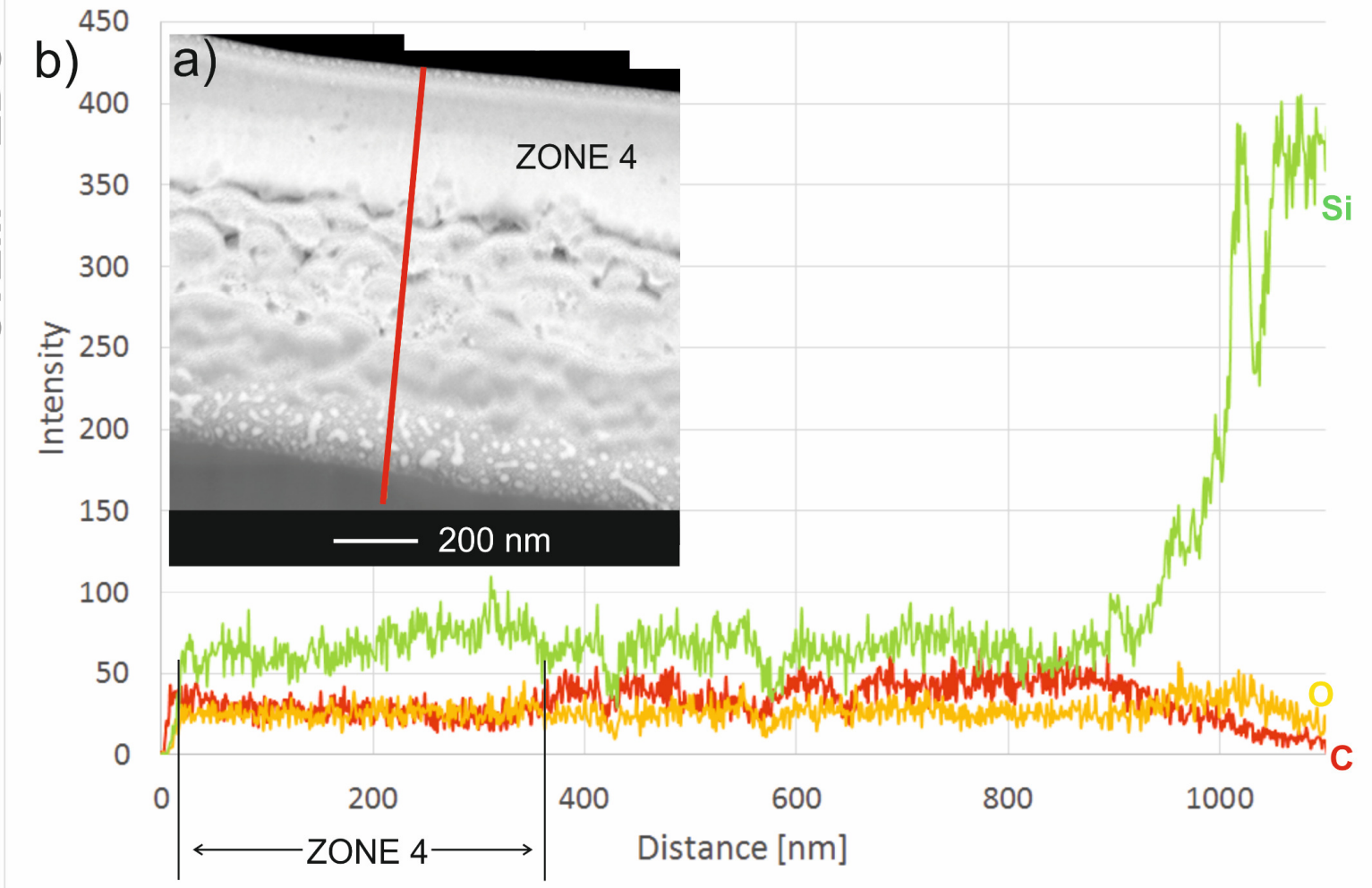
International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

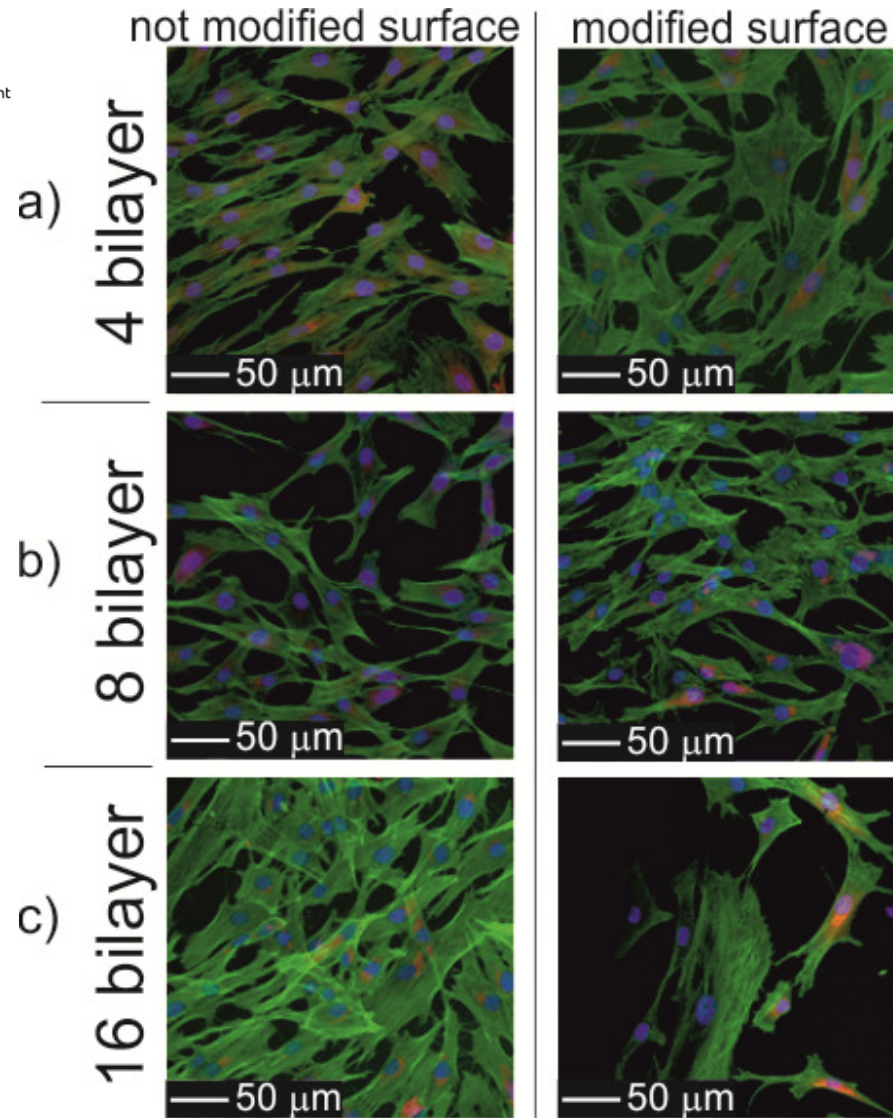
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STEM + EDS







- Project NCN nr: 3066/B/T02/2011/40- *FINISHED*
- Project NCN nr: 2012/06/M/ST8/00408- *HARMONIA- FINISHED*
- Project NCN nr: 2012/07/B/ST8/03396- *OPUS- FINISHED*
- Project NCN nr: 2014/15/B/ST8/00103- *OPUS- FINISHED*
- Project NCN nr: 2015/19/B/ST8/00942- *OPUS- in progress*
- Project NCBR, number: DZP/M-ERA.NET-2015/285/2016- *in progress*



Title: Bio-compatible, wear resistant, decorative coatings for biological, corrosive fluids interaction- development and their multiscale research

Project WND-POWR.03.02.00-00-1043/16

International interdisciplinary PhD Studies in Materials Science with English as the language of instruction

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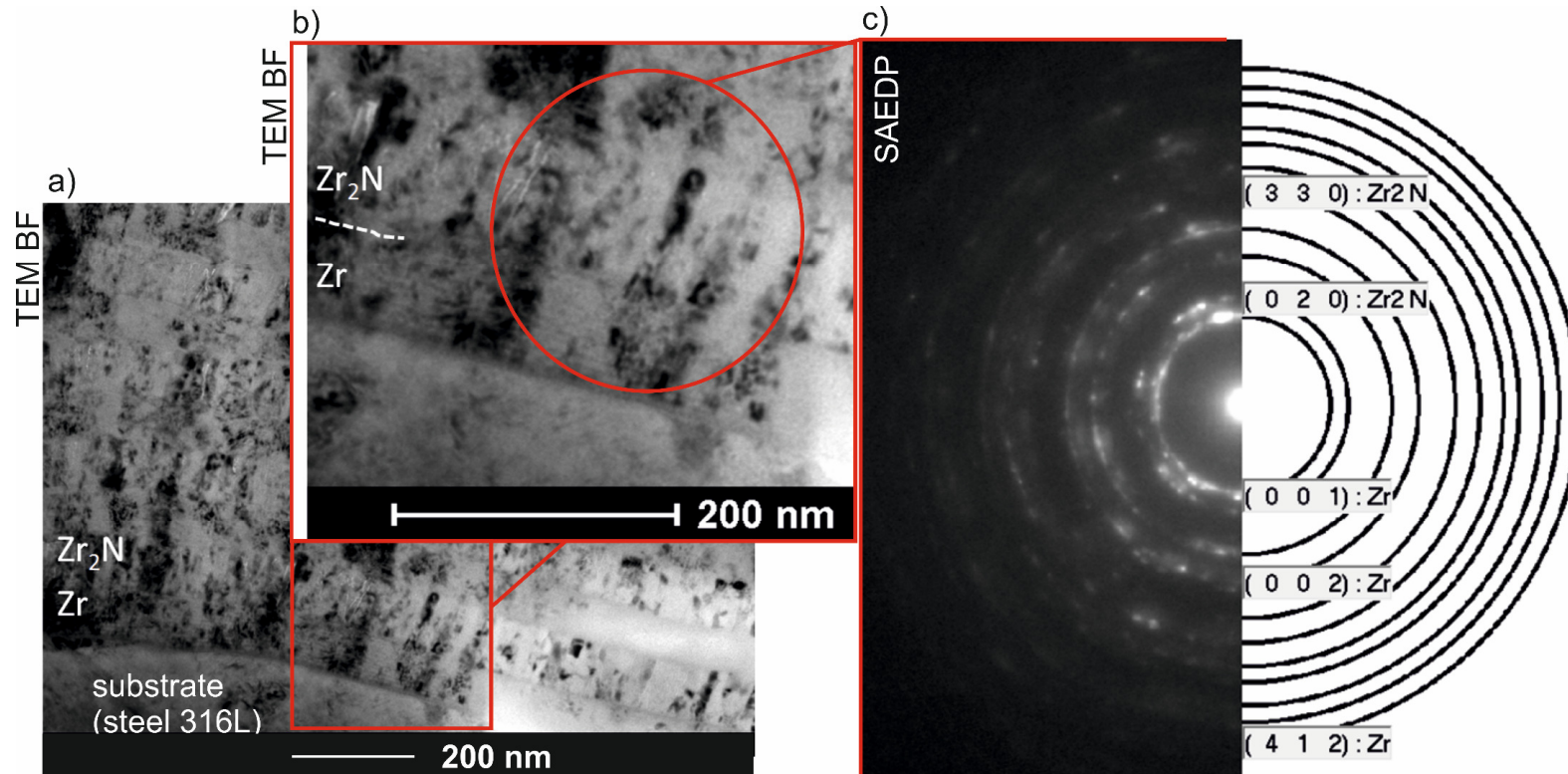


Multilayer Zr/Zr₂N coatings

As deposited coating (Before mechnaical tests)
Microstructure characterization



Microstructural characterization of the as-deposited coatings by TEM

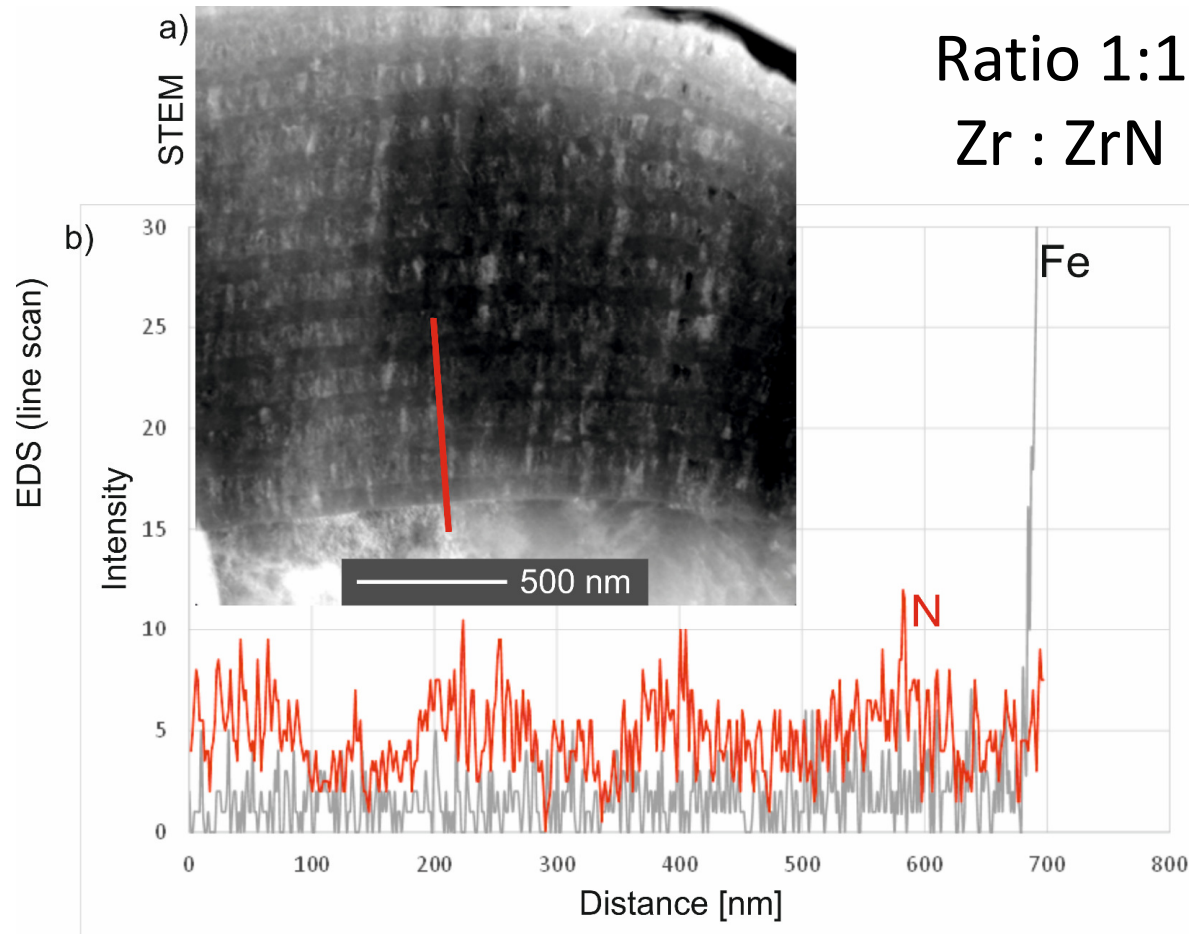


Ratio 1:1

Zr:ZrN

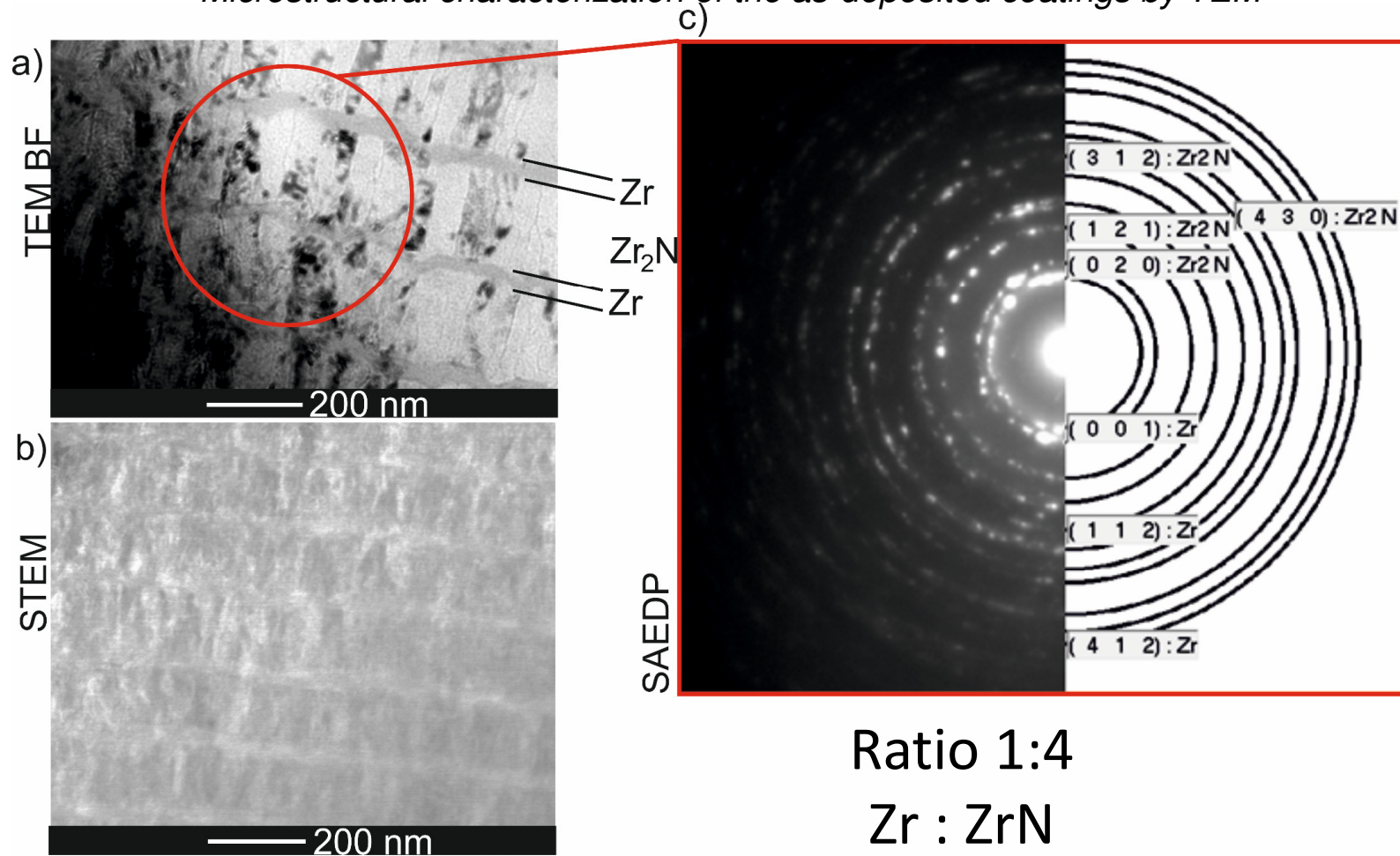


Microstructural characterization of the as-deposited coatings by TEM





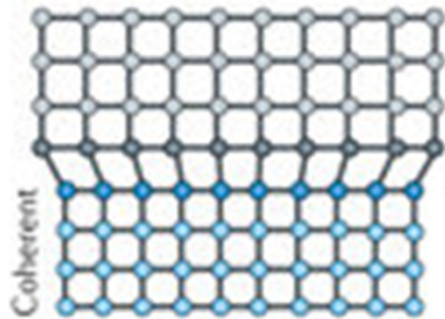
Microstructural characterization of the as-deposited coatings by TEM



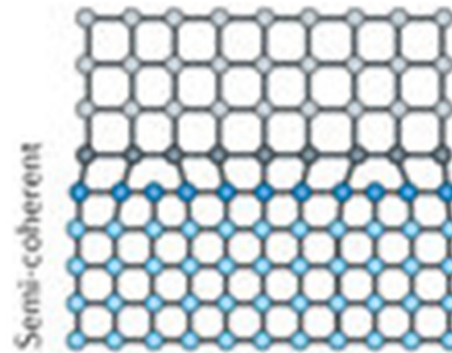


Microstructural characterization of the as-deposited coatings by TEM

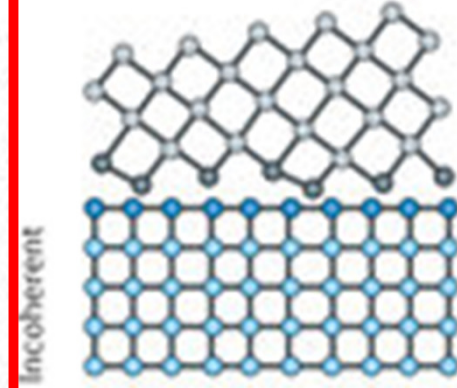
The coherent interface



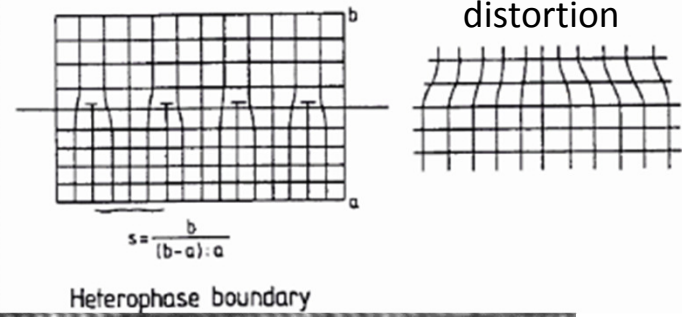
The semi-coherent interface



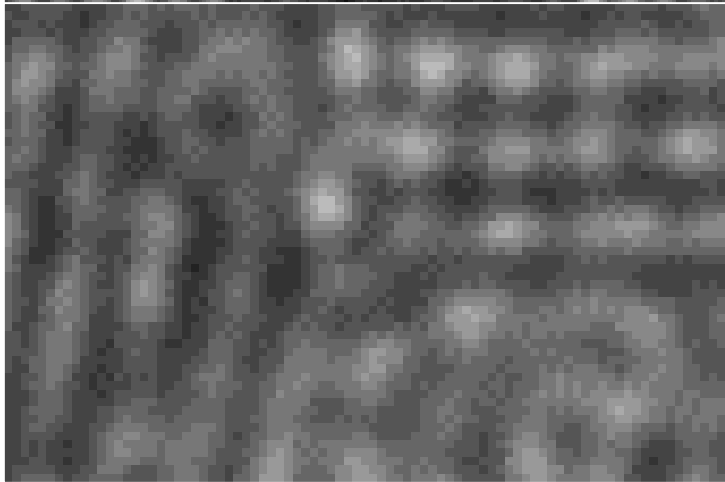
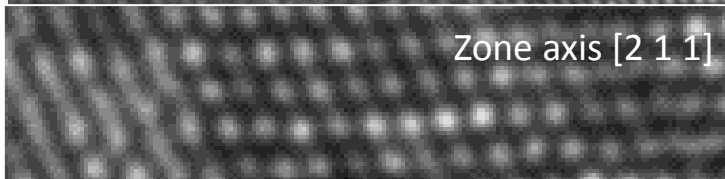
The incoherent interface



Coherency in between Zr and Zr_xN

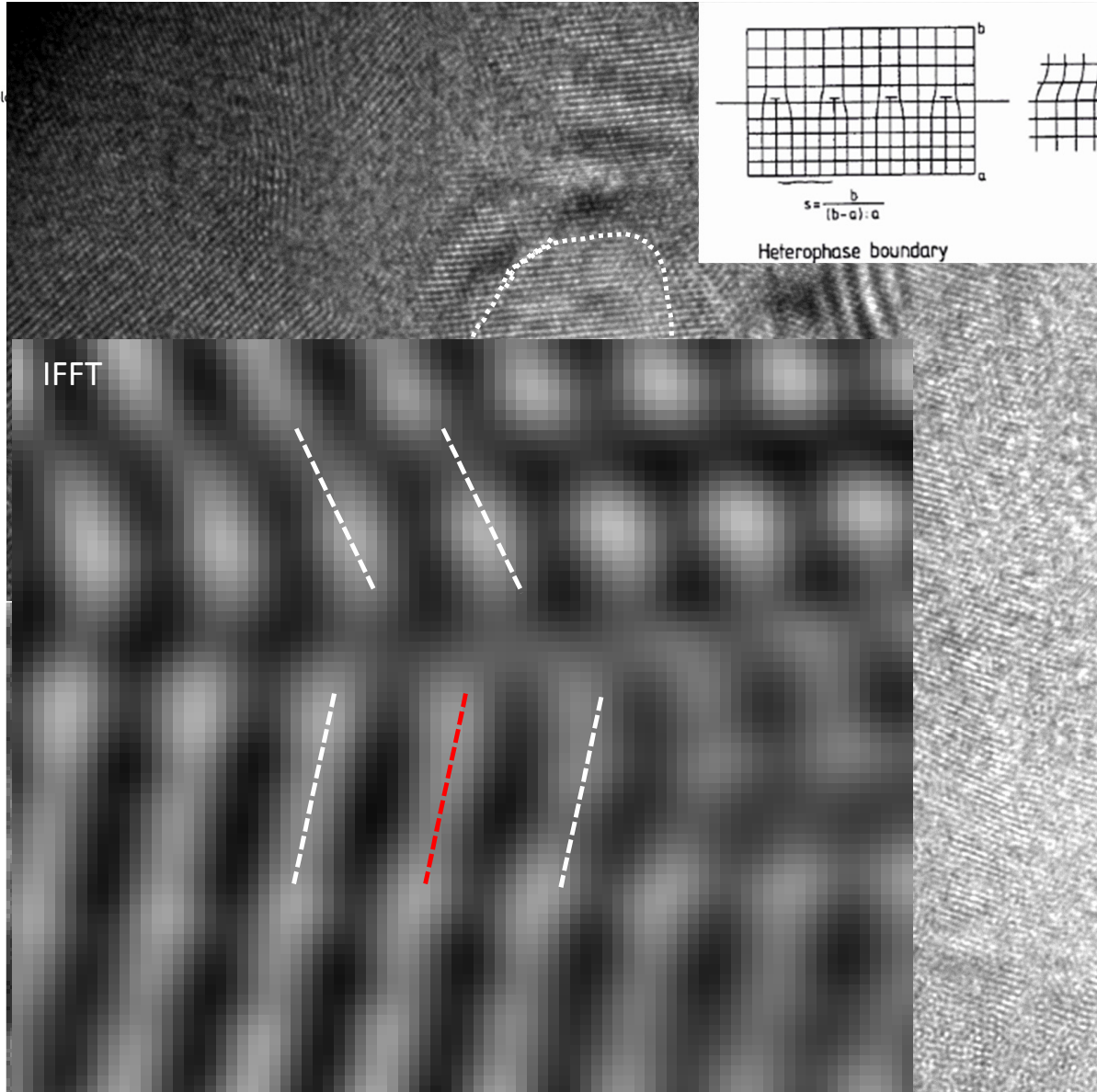


HRTEM



5 nm





HRTEM



Multilayer Zr/Zr₂N coatings

Coating after mechanical tests
Microstructure characterization

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Micromechanical tests results

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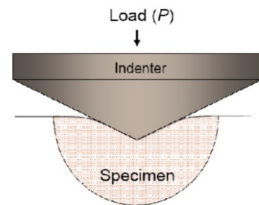
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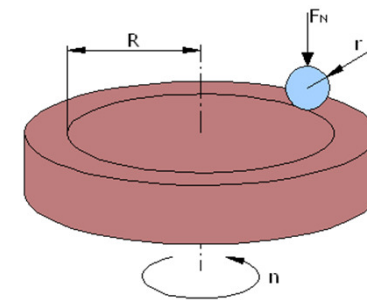
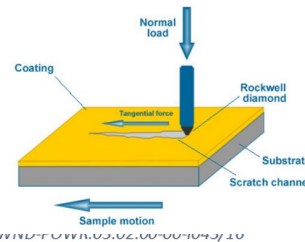
Micromechanical tests results

coating	Indentation		Critical upload		Tribological tests results		
	H [GPa]	E [GPa]	Lc1 [N]	Lc2 [N]	Coefficient of wear under F=0,25N mm ³ /Nm	Coefficient of friction	Number of cycles to coating remove under the F=1N
ZrN reference	29,95	375	4.9	14.6	$2.92 \cdot 10^{-6}$	0.18	8200
Zr/ZrN Ratio 1:1	11,14	158	0.9	17.5	$15.55 \cdot 10^{-6}$	0.12	100
Zr/ZrN Ratio 1:2	12,43	200	1.7	28.4	$5.10 \cdot 10^{-6}$	0.1	280
Zr/ZrN Ratio 1:4	14,59	221	1	> 30	$6.74 \cdot 10^{-6}$	0.1	750

Indentation test (Hardness test)



Scratch test set-up



- **Hardness – resistance to penetration of a hard indenter** *3D Studies in Materials Science with English as the language of instruction*

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Microstructural characterization of the coatings by TEM after the wear test

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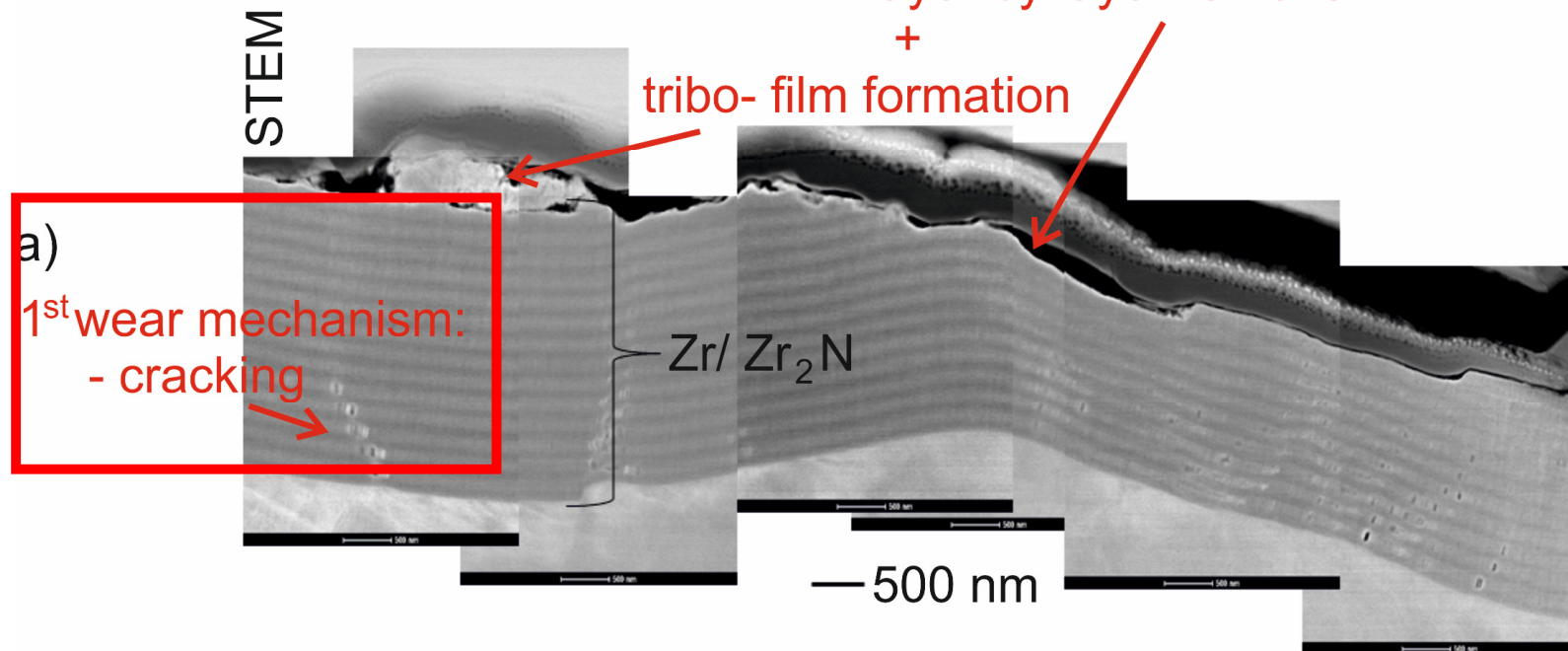


Microstructural characterization of the coatings by TEM after the wear test

b) 2nd wear mechanism:
- layer by layer remove
+

tribo- film formation

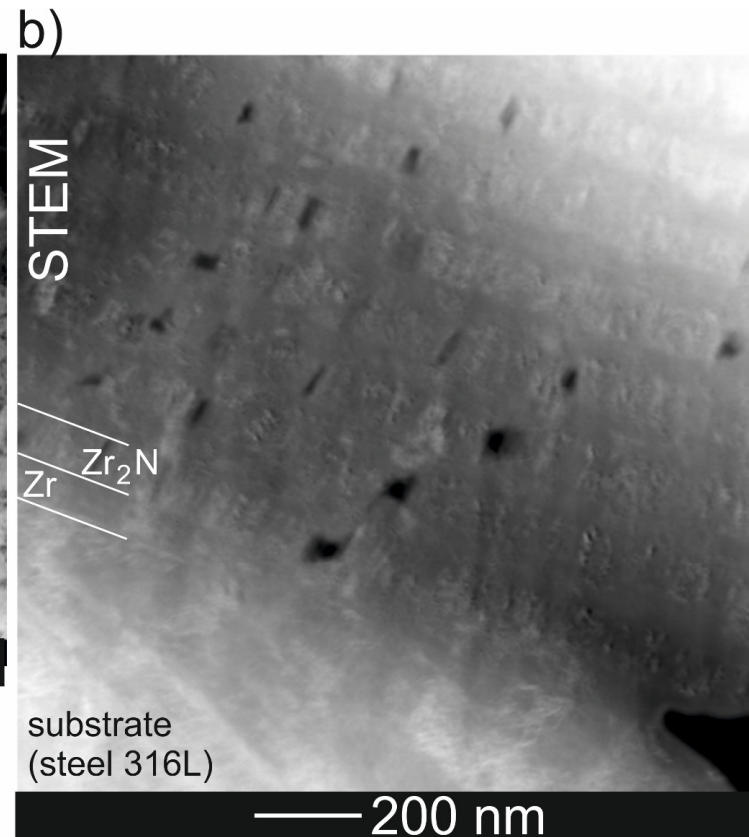
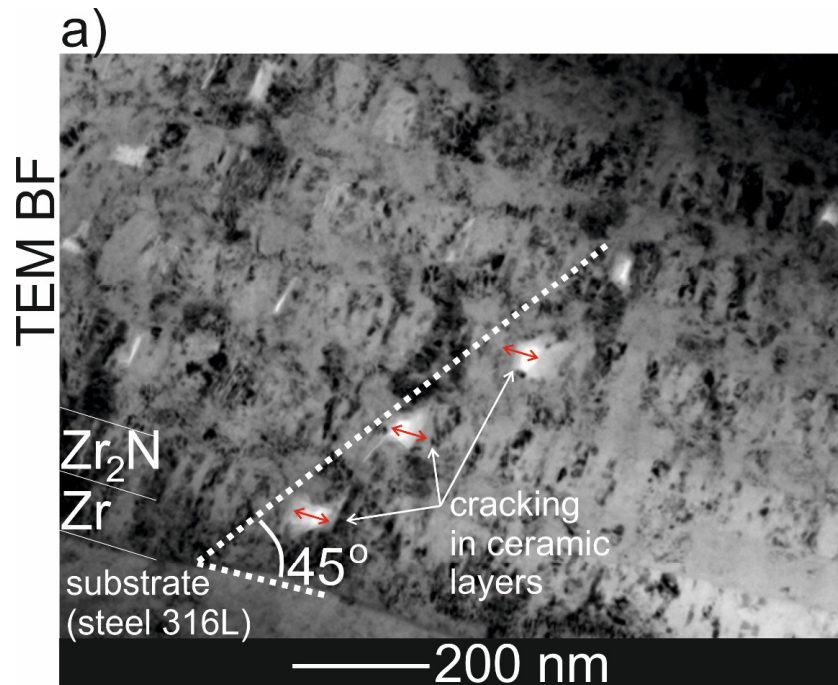
a) 1st wear mechanism:
- cracking



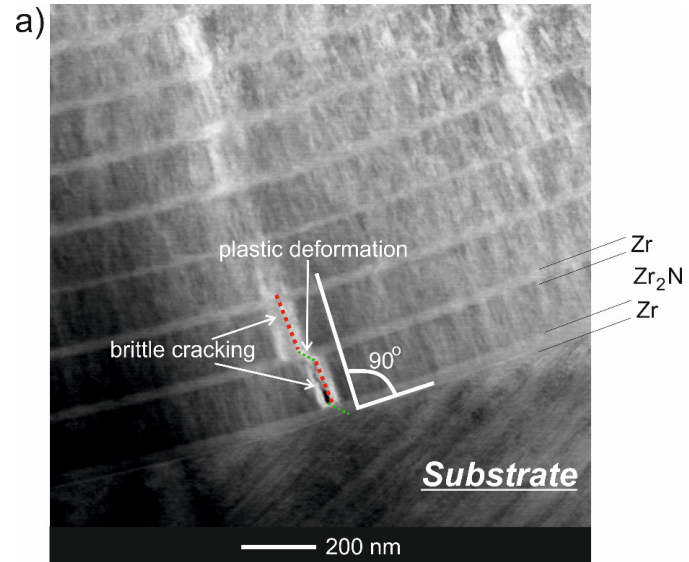
Ratio 1:1
Zr : ZrN



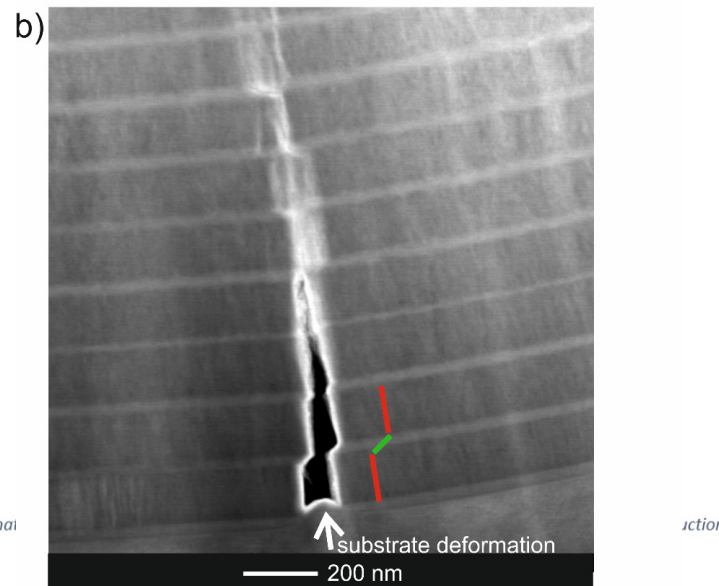
Microstructural characterization of the coatings by TEM after the wear test



Ratio 1:1
Zr : ZrN

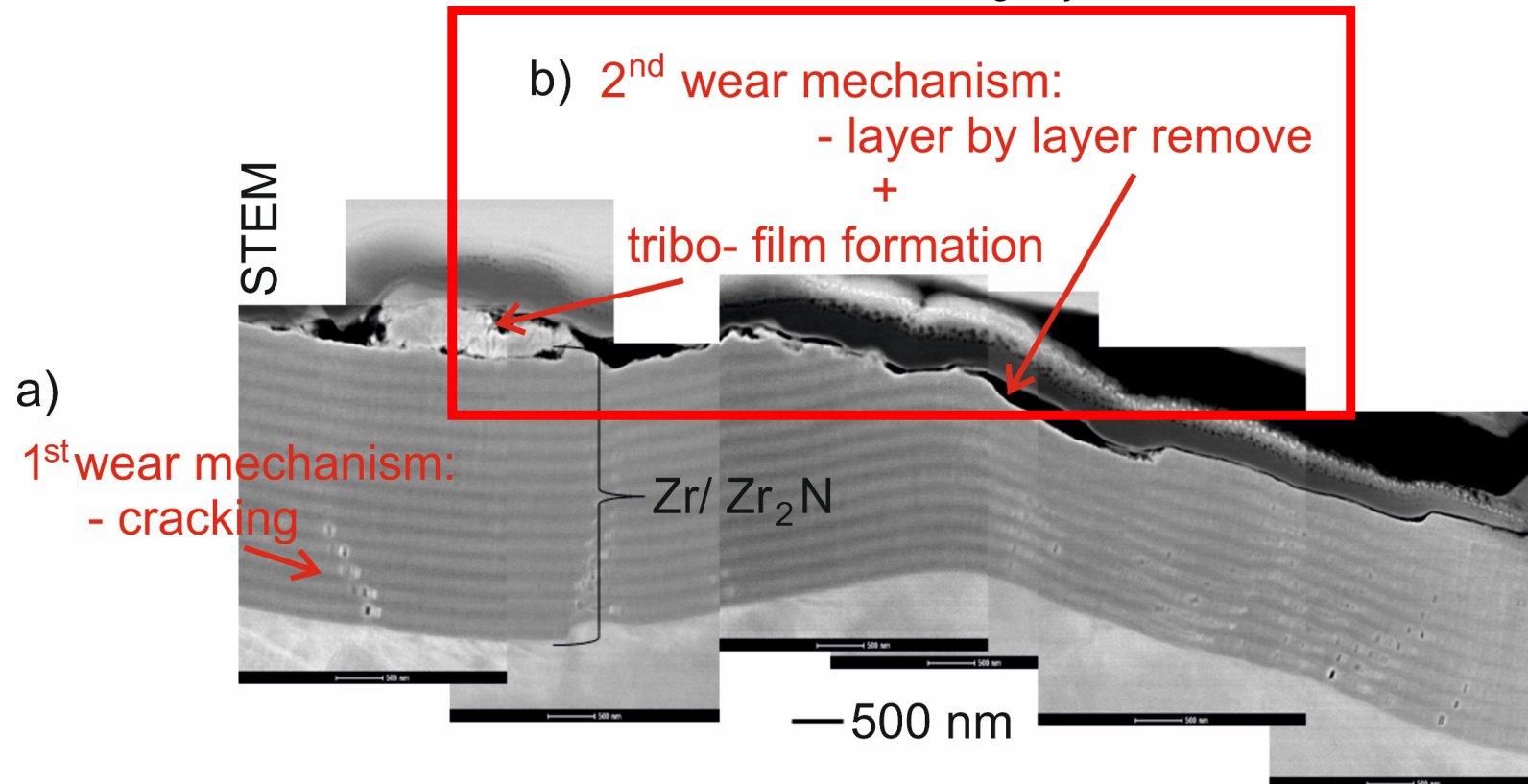


Ratio 1:4
Zr : ZrN



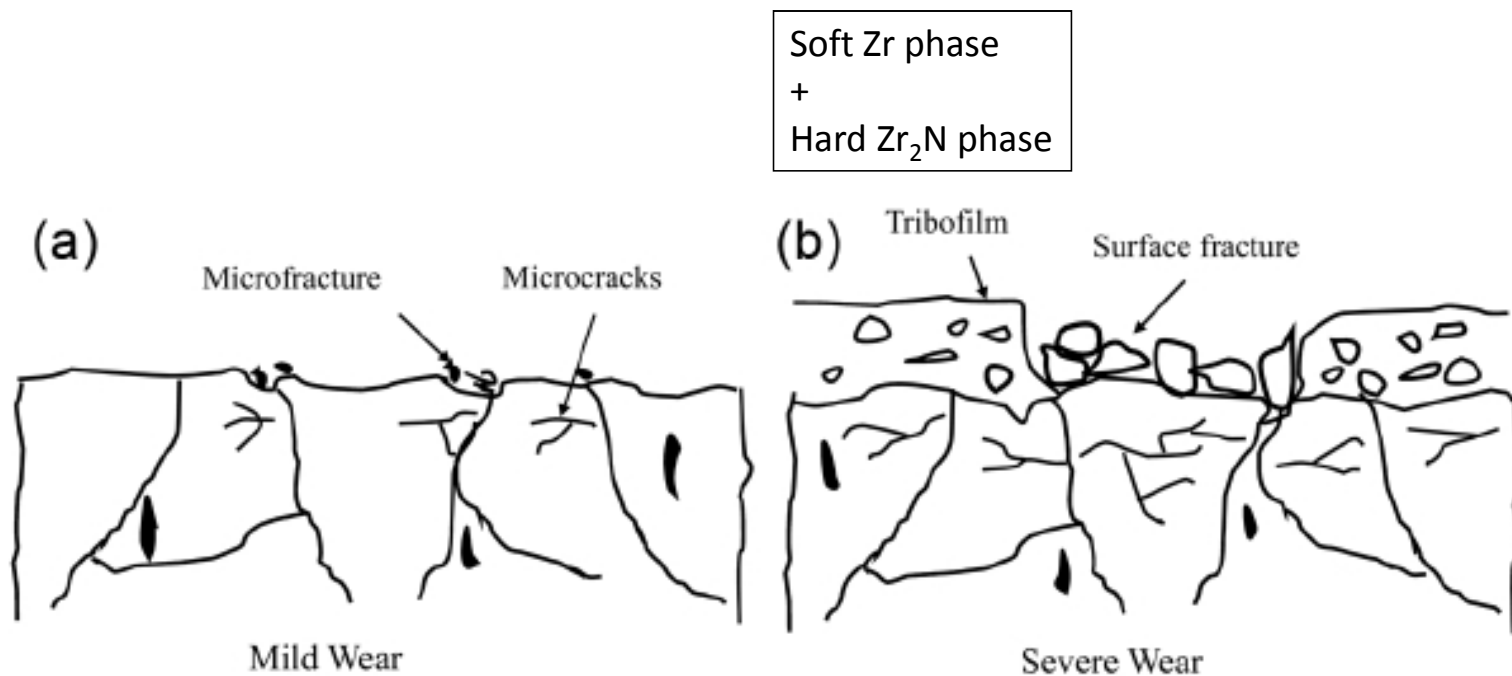


Microstructural characterization of the coatings by TEM after the wear test



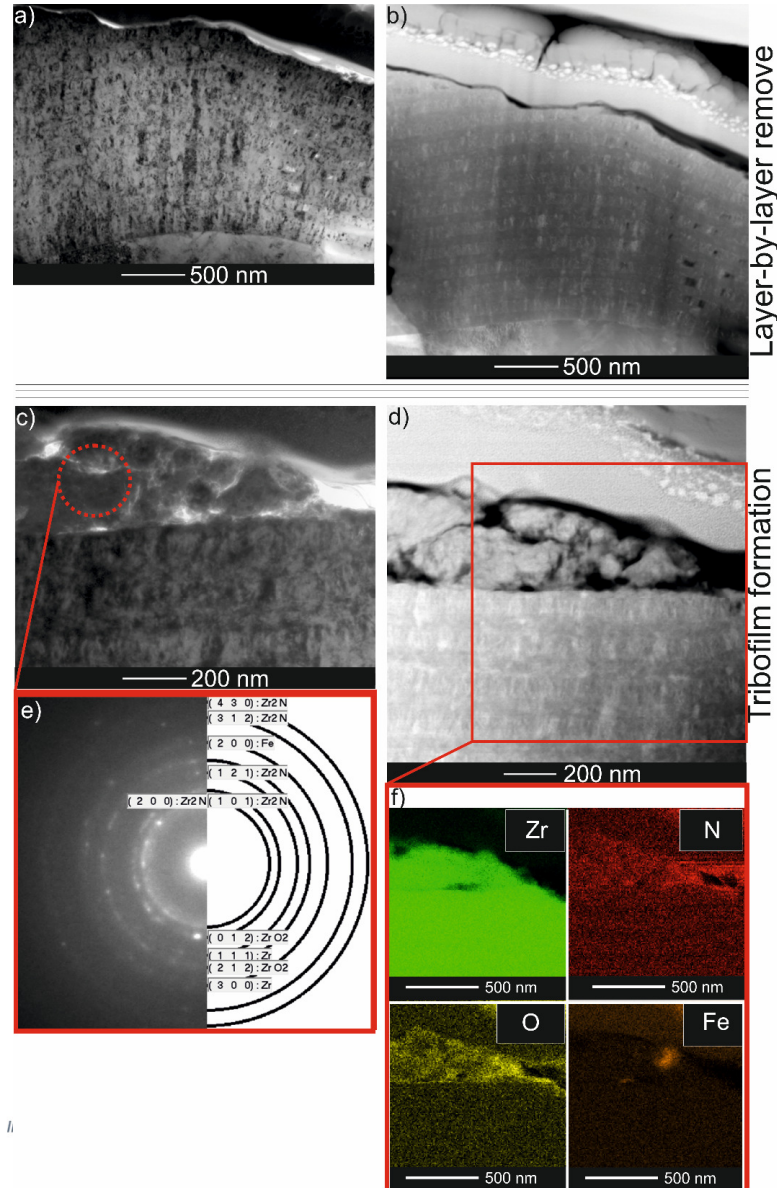


Microstructural characterization of the coatings by TEM after the wear test



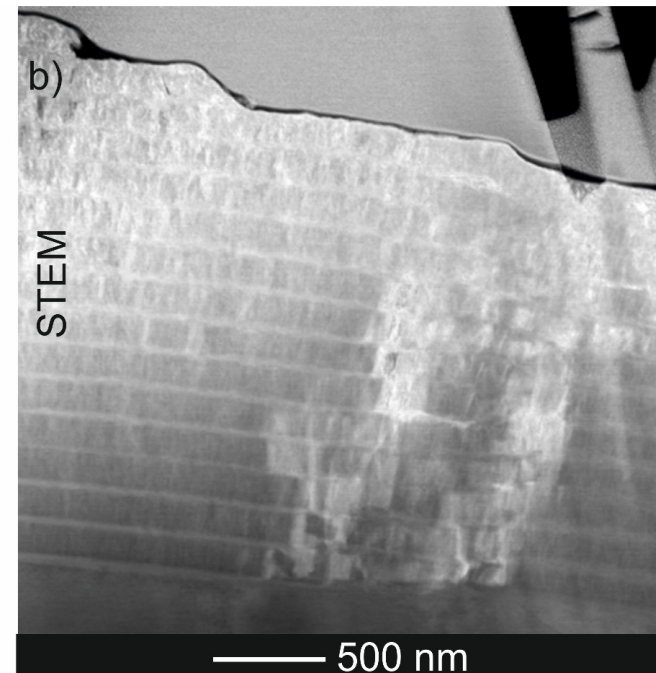
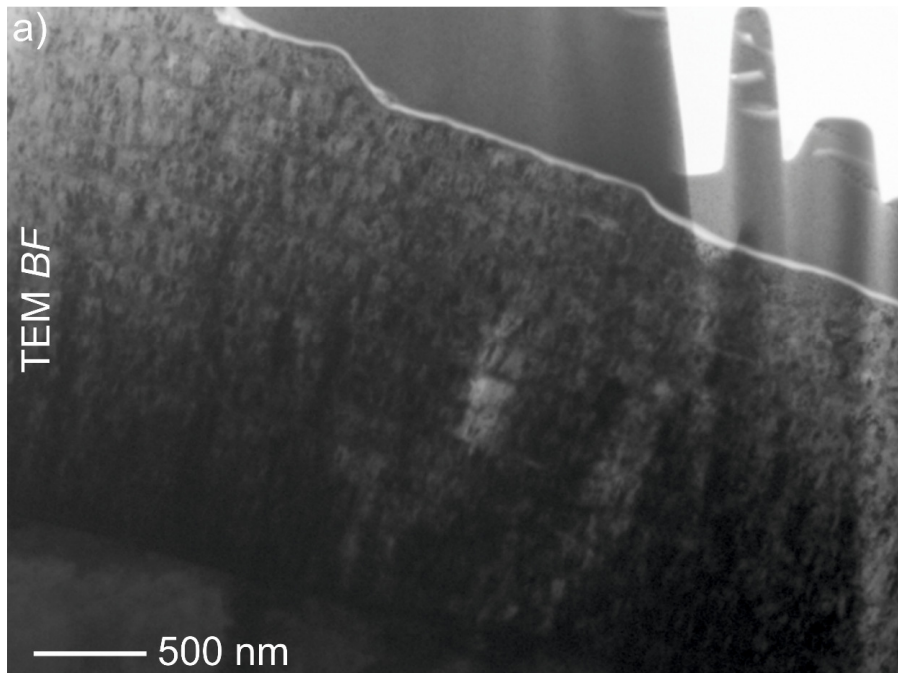


Ratio 1:1
Zr : ZrN





Microstructural characterization of the coatings by TEM after the wear test



Ratio 1:4
Zr : ZrN



Mechanical in situ tests performed in the SEM chamber

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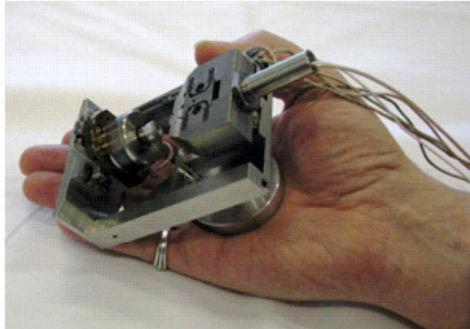
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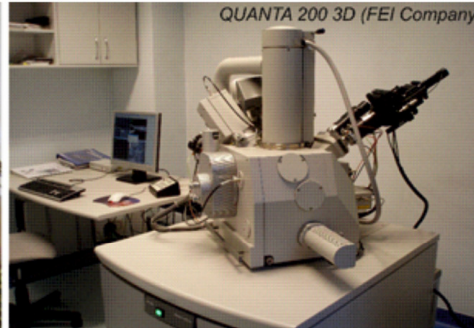


Mechanical in situ tests performed in the SEM chamber

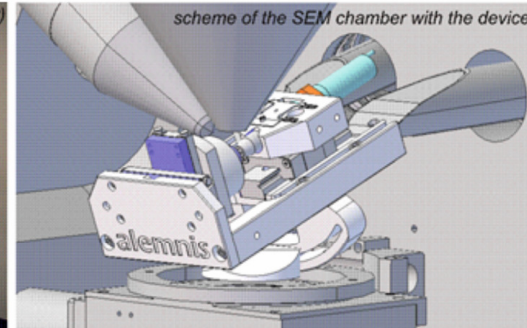
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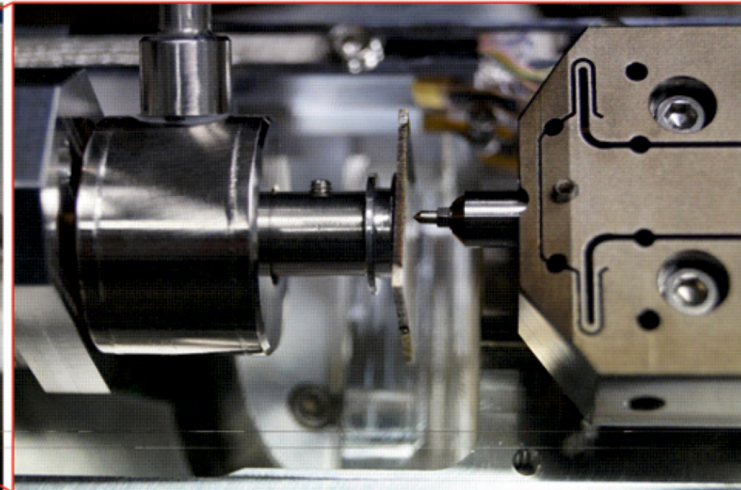
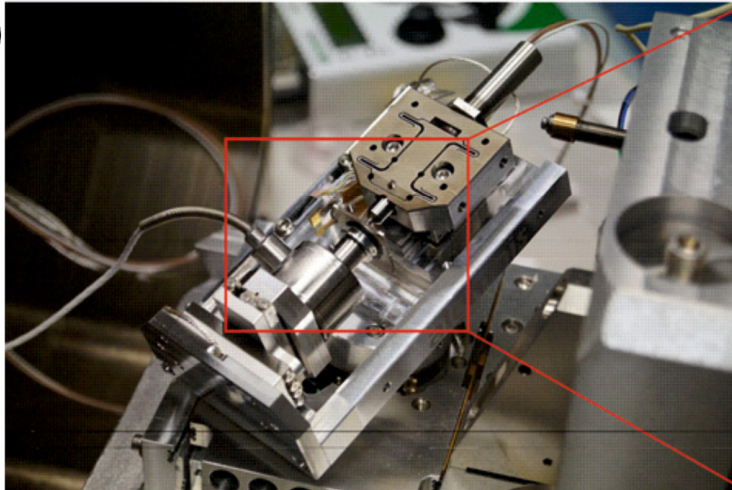
b)



c)



d)



e)

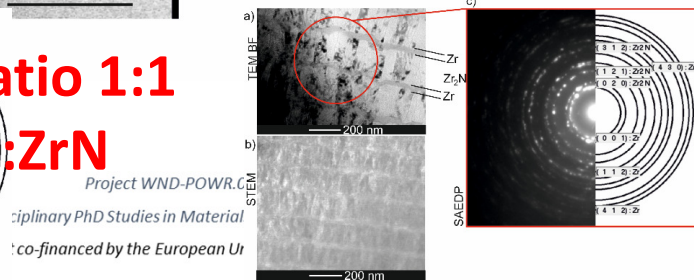
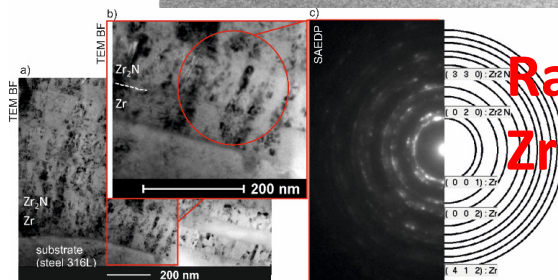
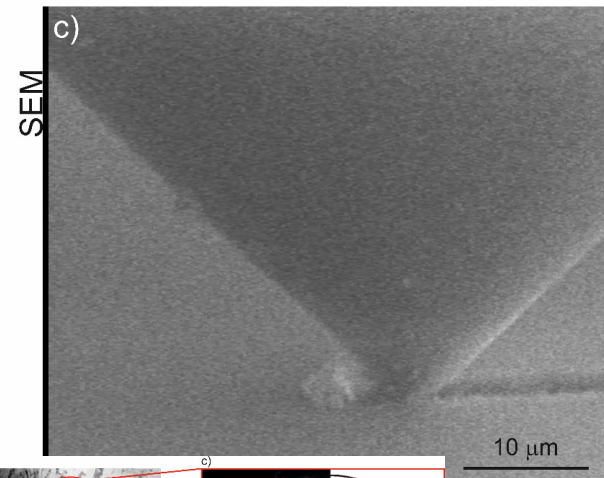
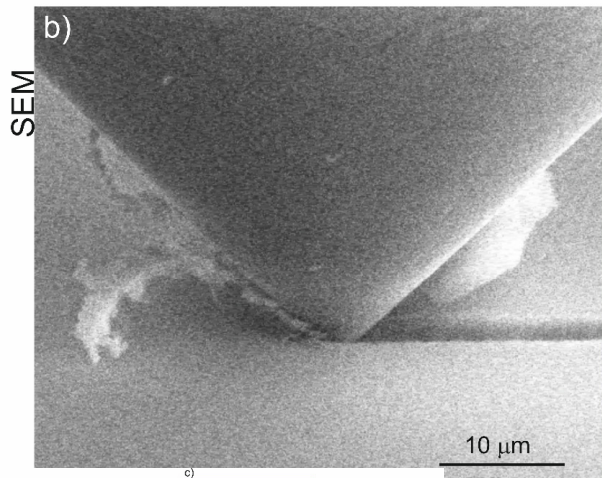
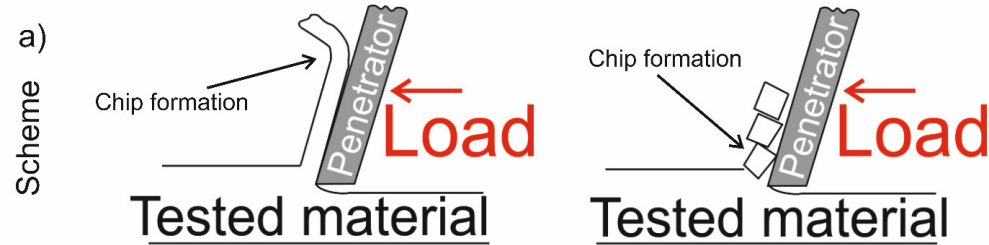
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Mechanical in situ tests performed in the SEM chamber



Project WND-POWR.C
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Multilayer Zr/Zr₂N coatings

Coating after corrosion tests
Microstructure characterization

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Corrosion is the destructive attack of a metal by its reaction with the environment

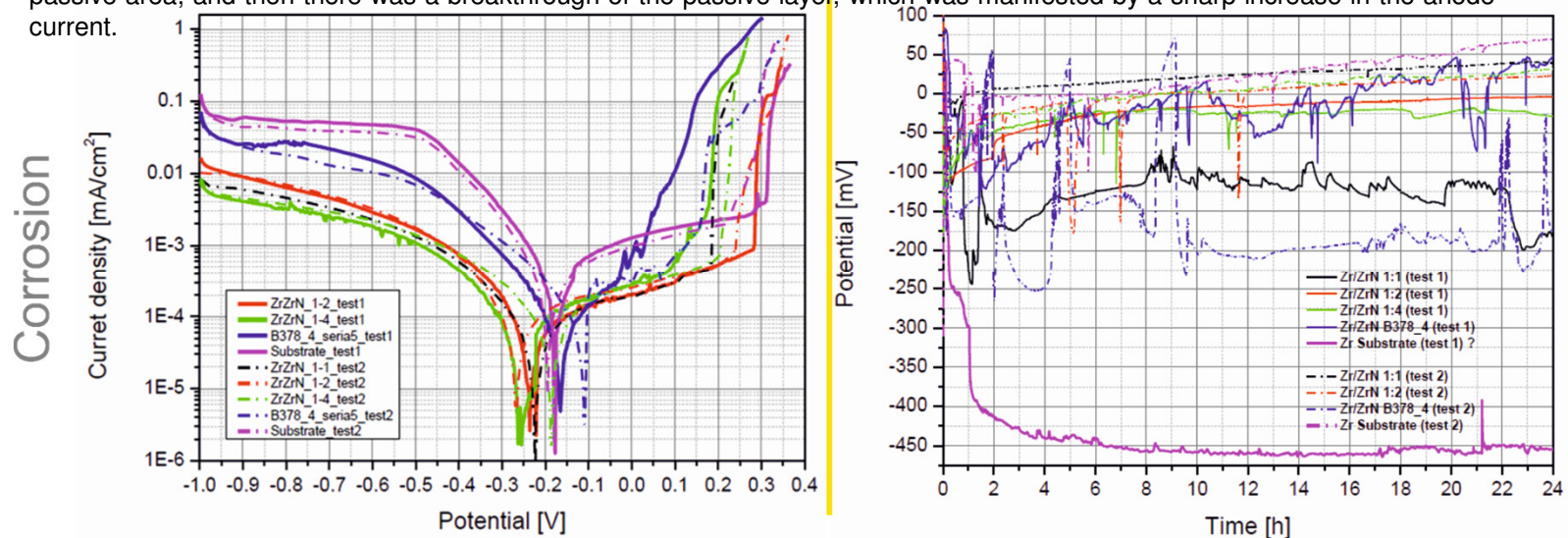
Corrosion test results

(Bio- corrosion in body fluids)



Linear Sweep Voltammetry – LSV

The nature of potentiodynamic curves indicates that the tested coatings and substrate (316L steel) underwent pitting corrosion in Ringer's solution. The sharp increase in the anode current, at potentials above 0 mV, indicates the formation of pitting on the surface of coatings and steel. In the cathodic area, the highest currents were recorded for the substrate (pink continuous and dotted curves). Coated substrates with all coatings showed much lower currents in the cathodic region, which indicated that the cathodic reaction (oxygen reduction reaction) was slower than on the uncoated substrate (316L). The course of the polarization curves in the anodic area of the uncoated substrate and of the samples with the coatings were very similar. They showed a passive area, and then there was a breakthrough of the passive layer, which was manifested by a sharp increase in the anode current.



The exception was the blue curve, recorded for sample B378 (with the carbon layer), on which a continuous increase in the anode current was visible without a clear passive area. Such character of the polarization curve indicated that the B378 sample exhibited the lowest corrosion resistance in the Ringer solution. Fig. 2 showed the corrosion potential changes registered within 24 hours in Ringer's solution. The corrosion potentials recorded for the sample Zr / ZrN_B378 (blue curves) deserve attention. They showed very large potential oscillations. In 24 hours it was impossible to achieve a steady state (stable value of corrosion potential). These results suggested that the sample showed the worst corrosion resistance in the Ringer fluid environment.



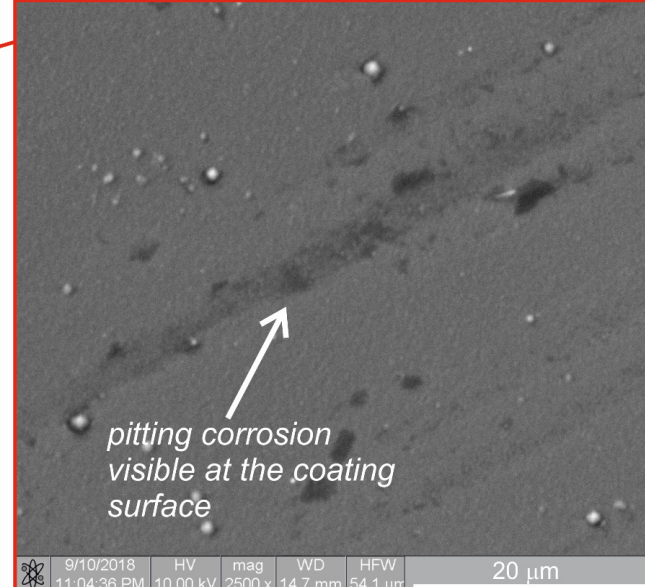
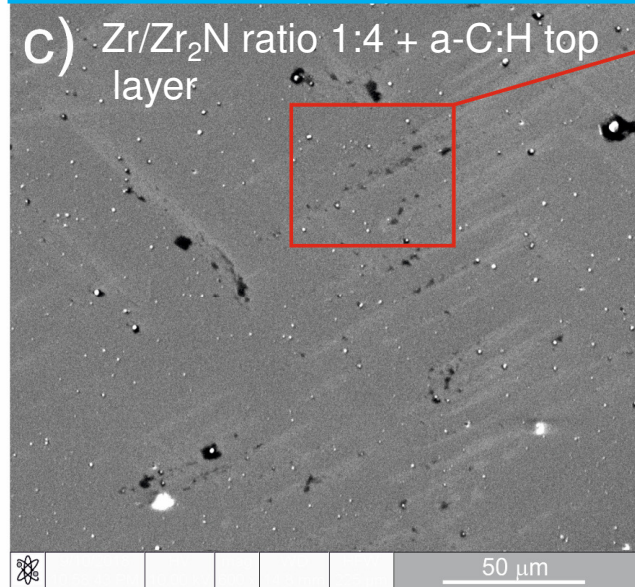
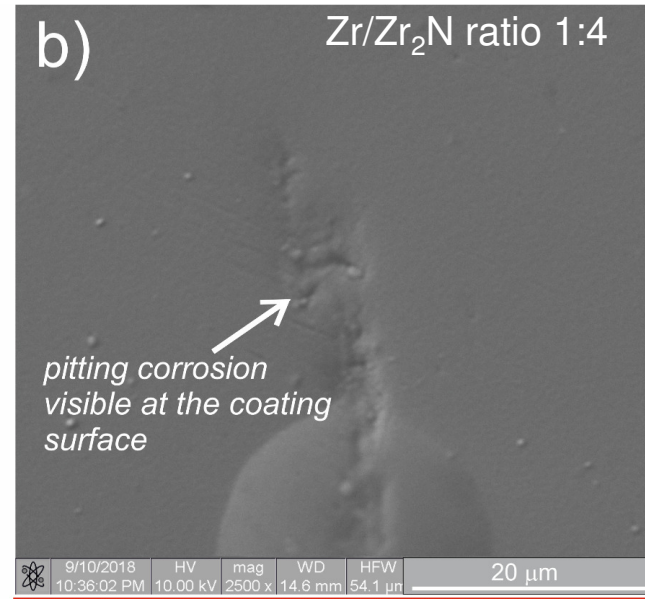
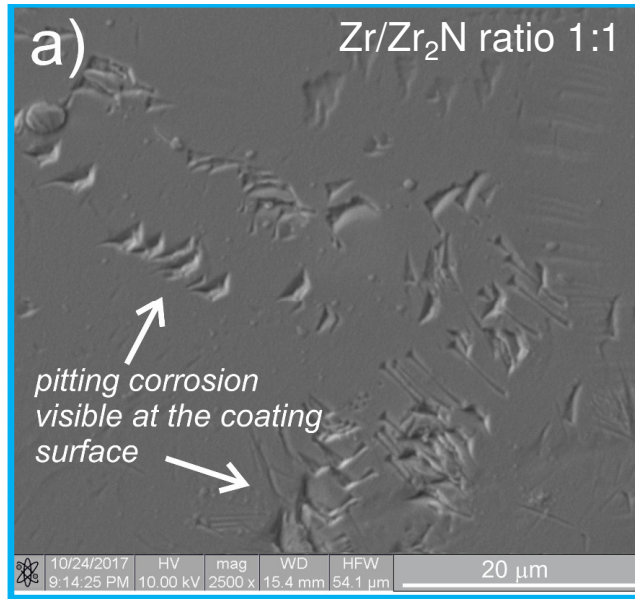
Microstructural characterization of the coatings by TEM after corrosion

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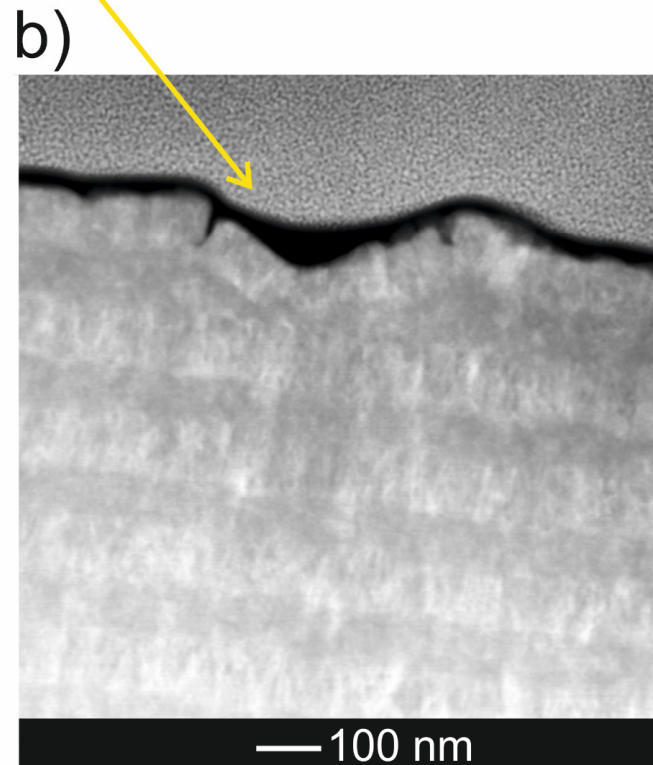
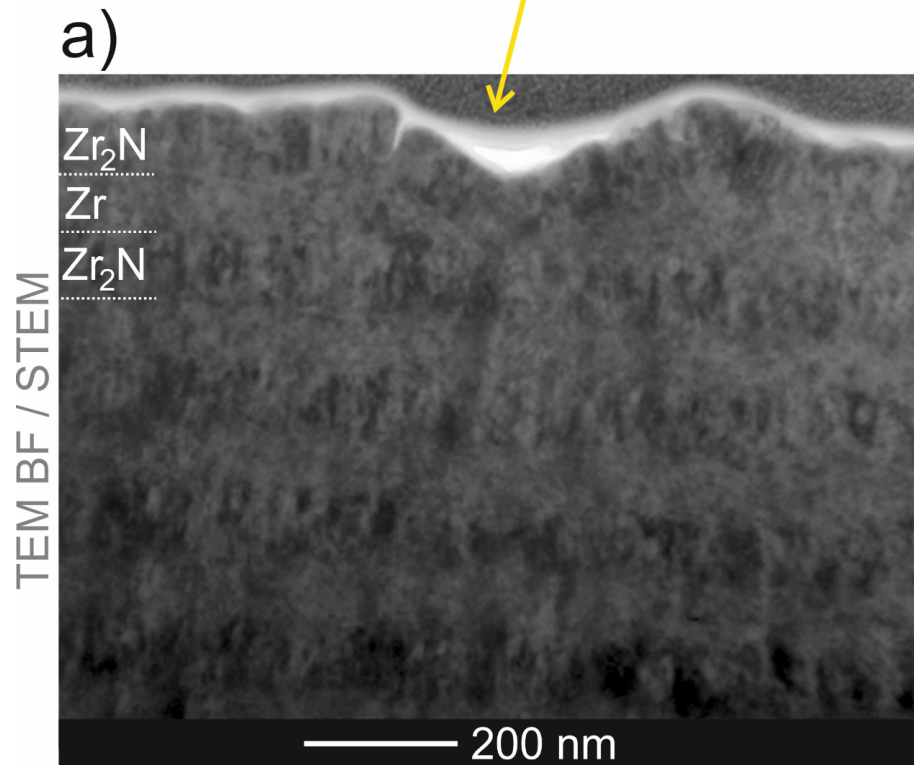
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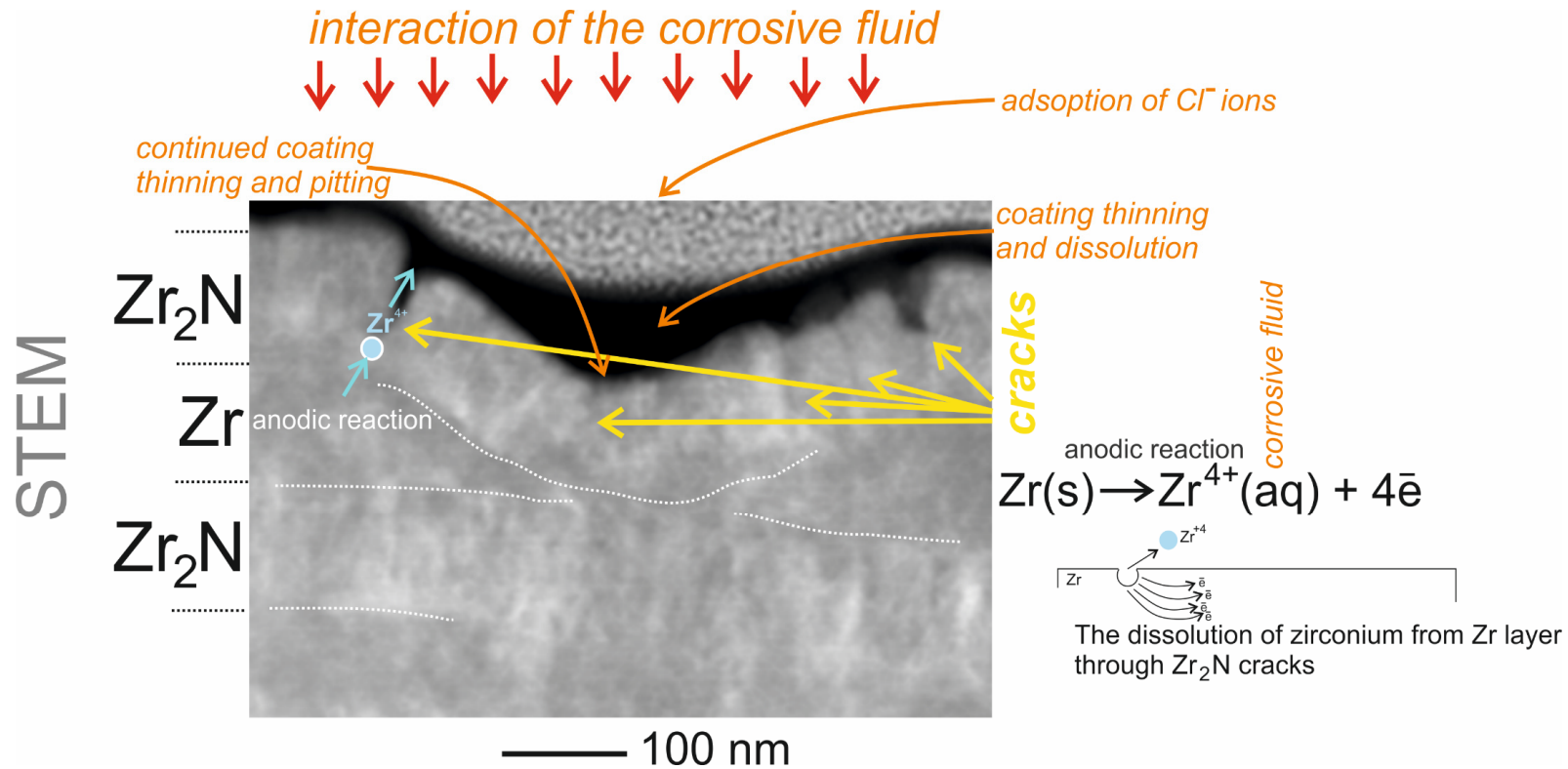
SEM





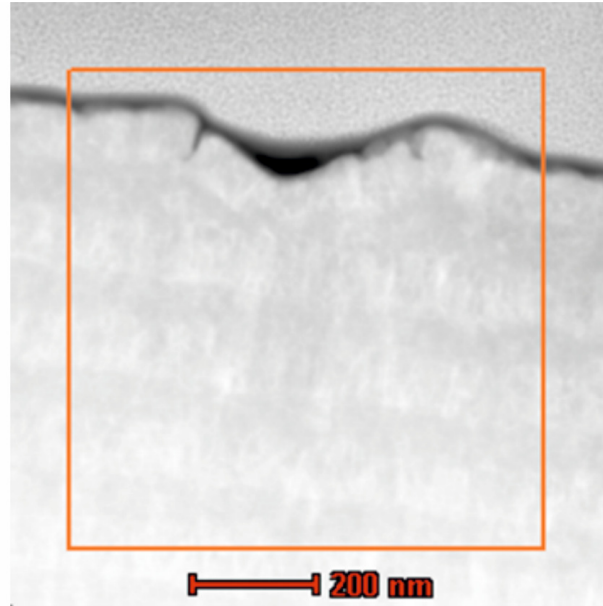
pitting corrosion visible at the cross-section



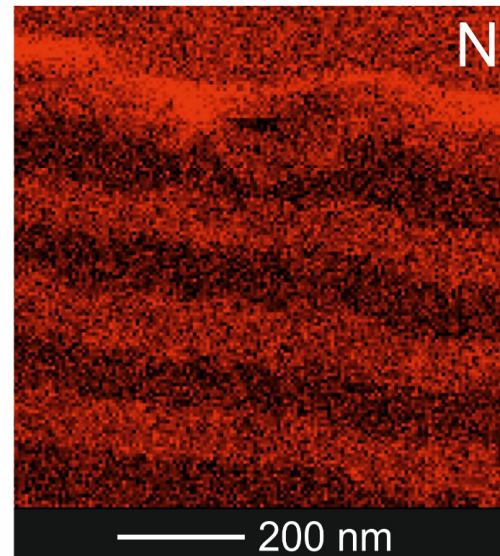
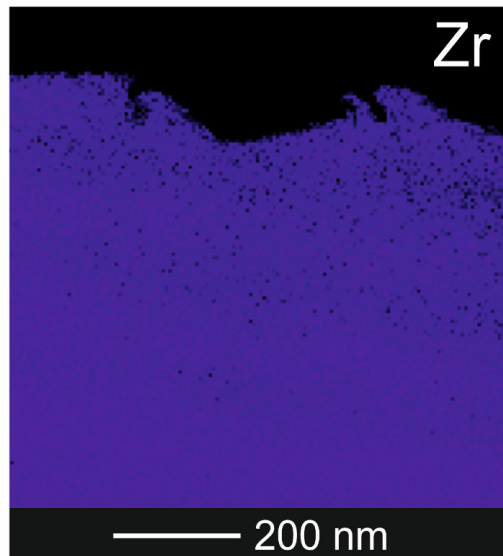




a)
STEM + EDS

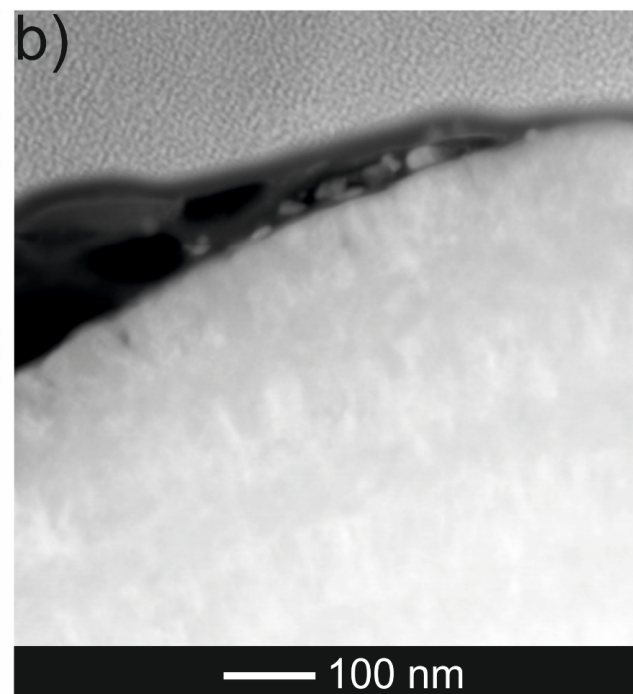
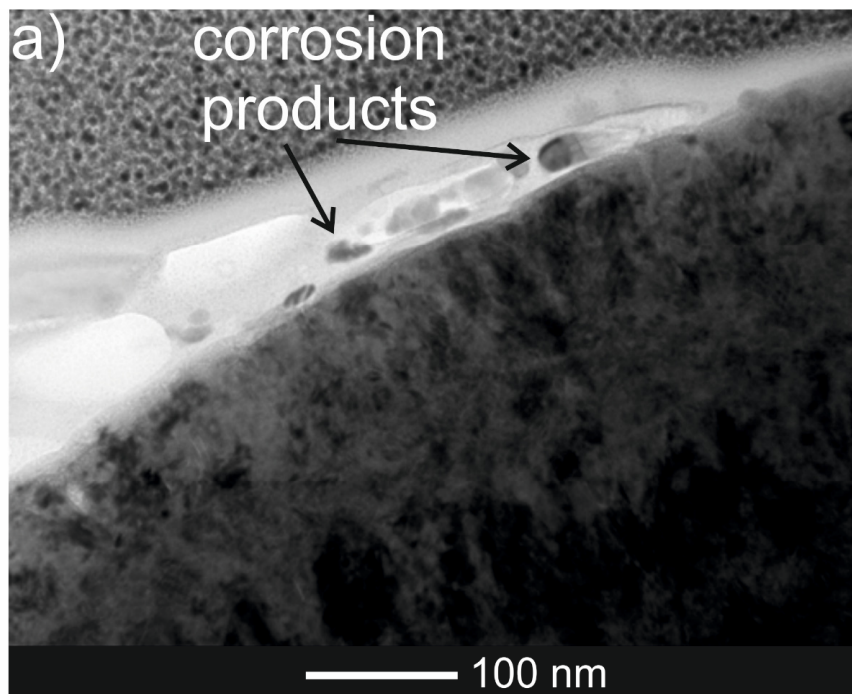


b)

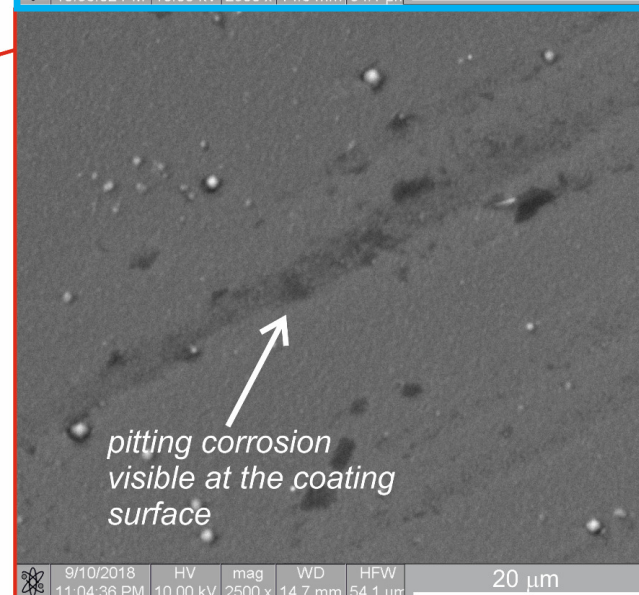
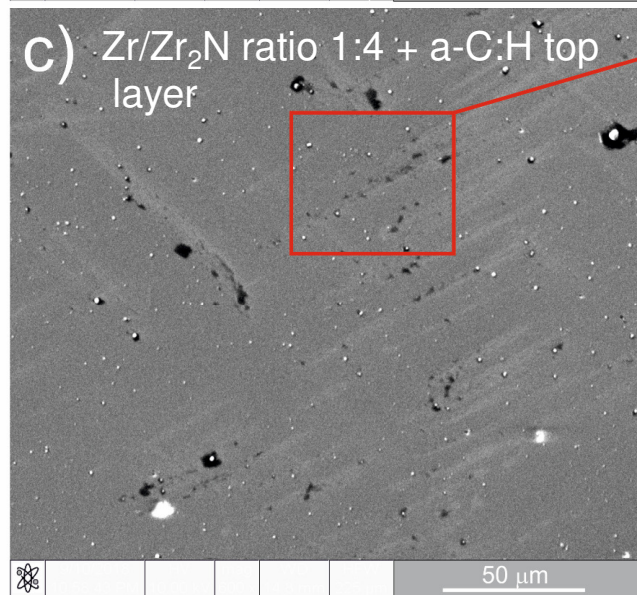
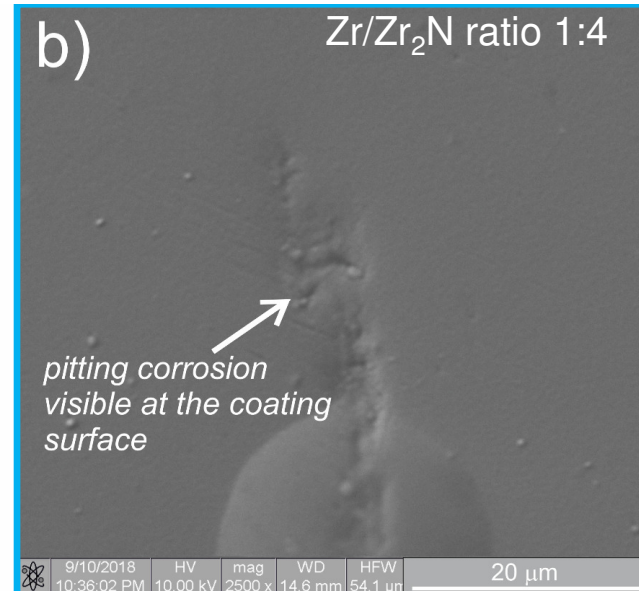
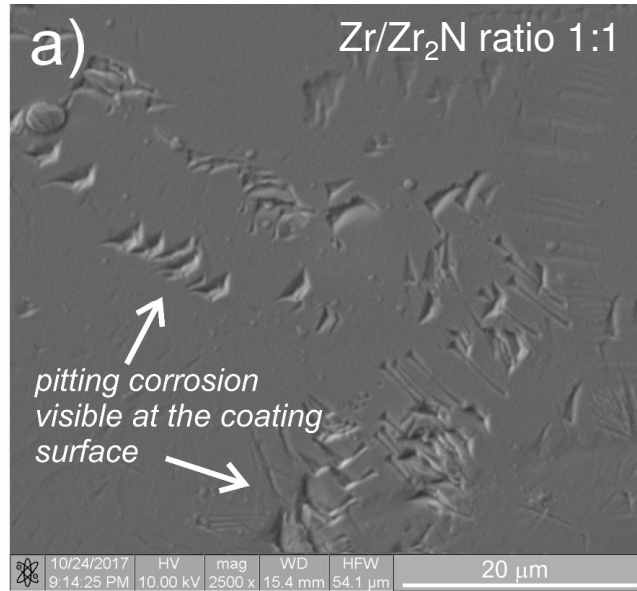




TEM BF / STEM

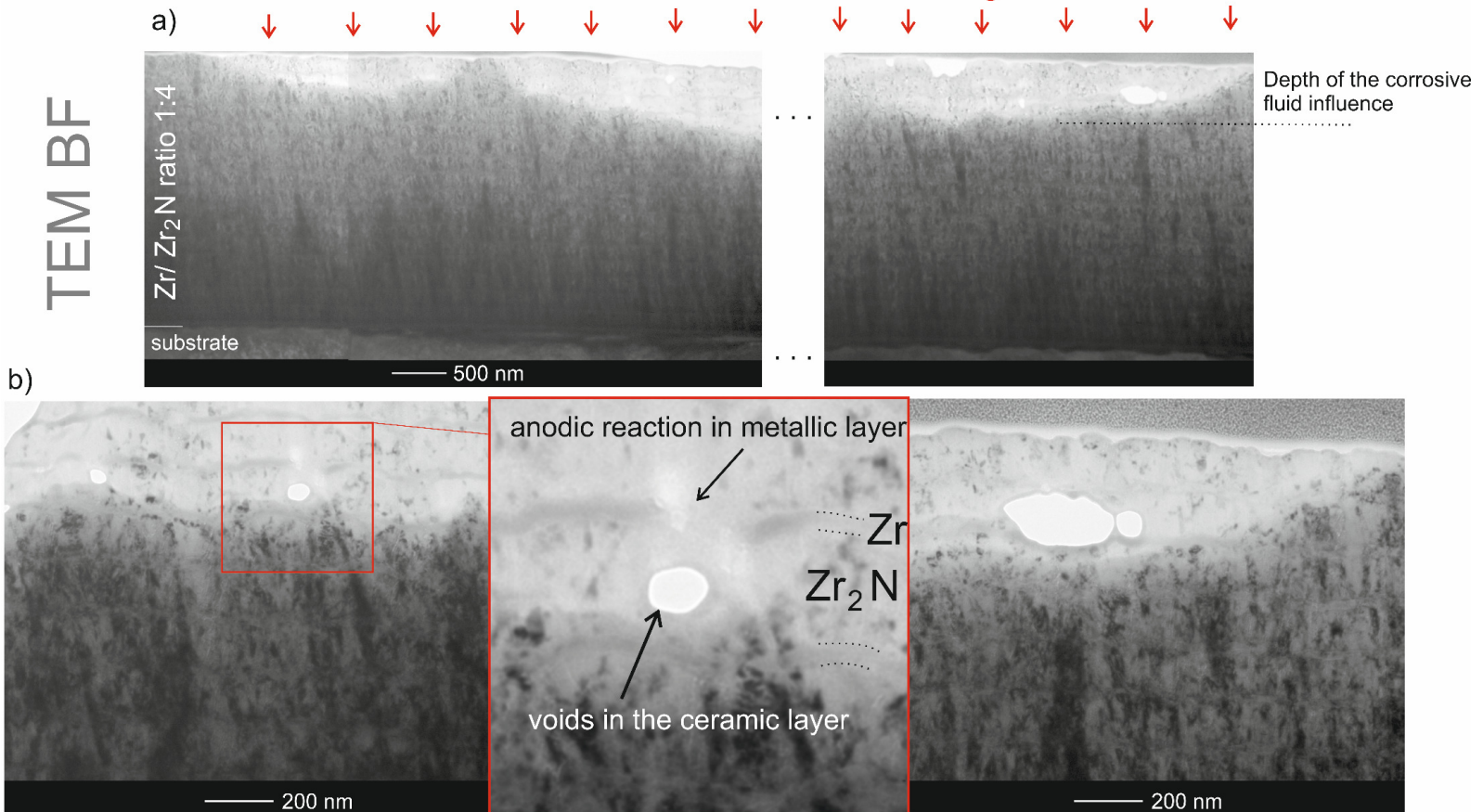


SEM





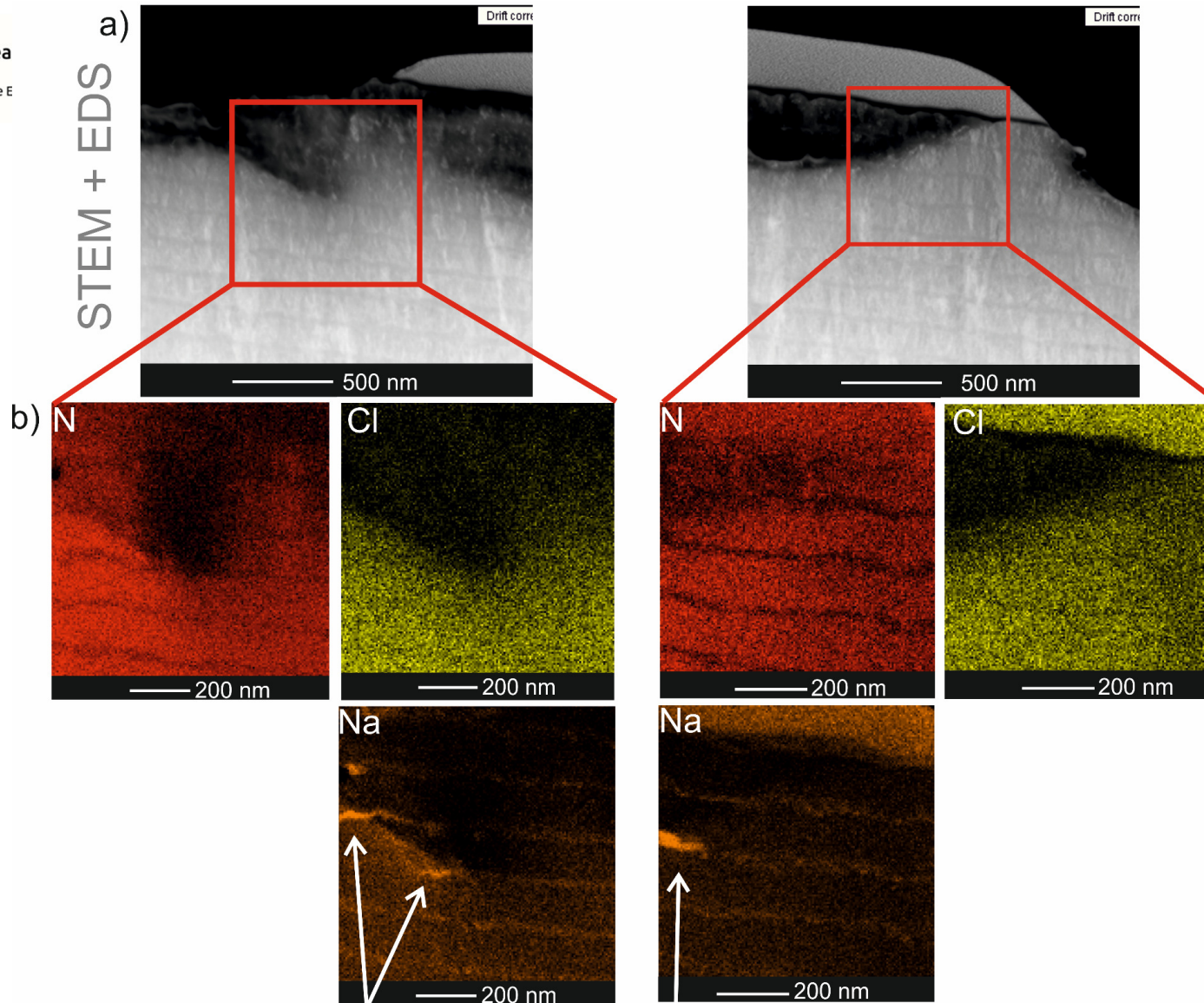
The influence of the corrosive fluid on the coating



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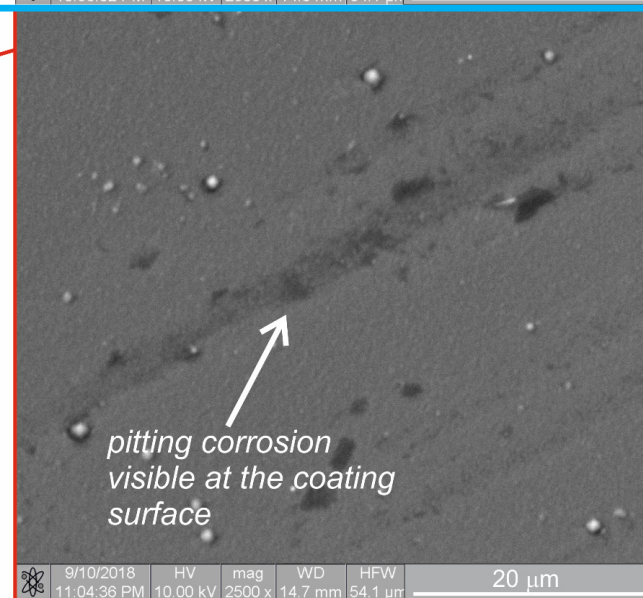
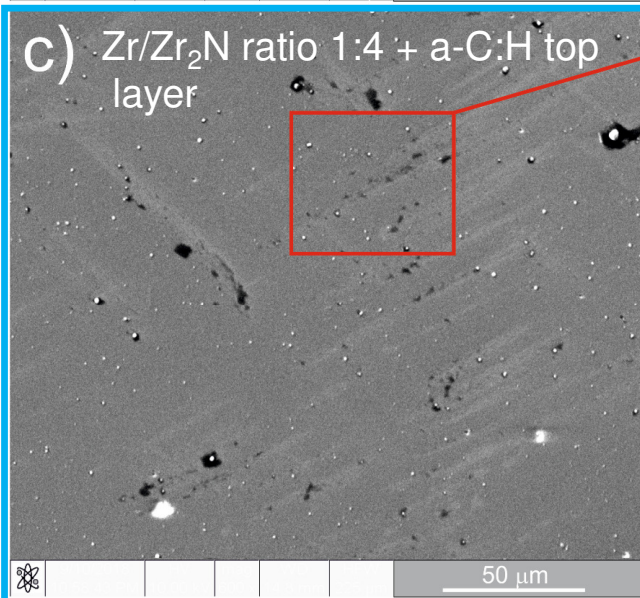
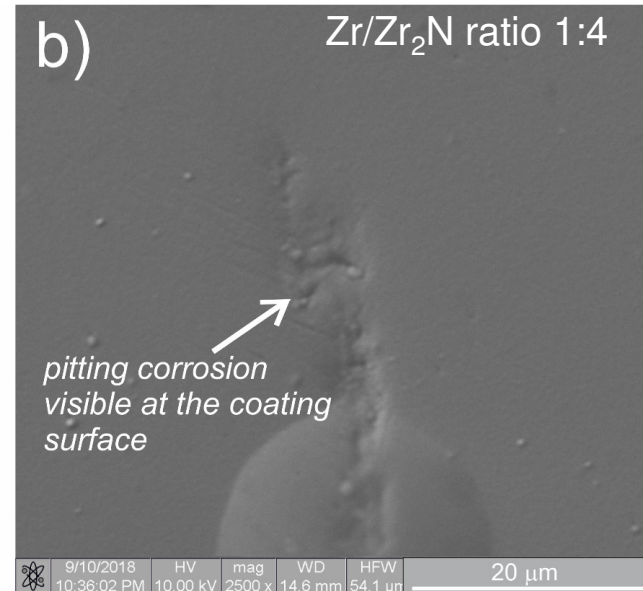
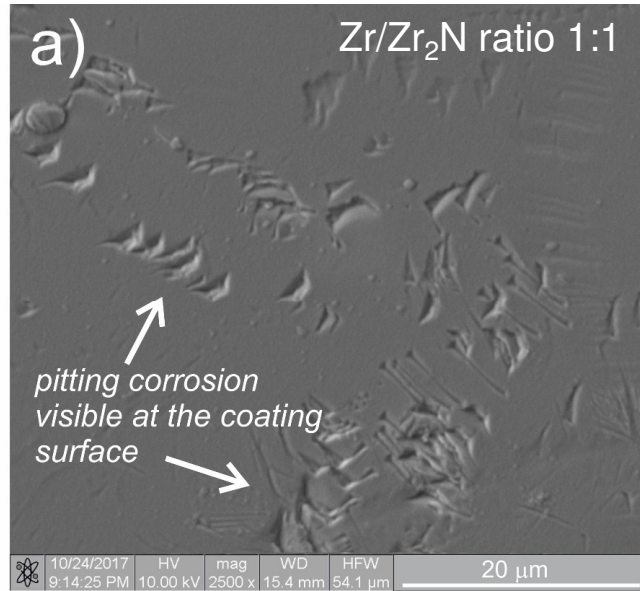
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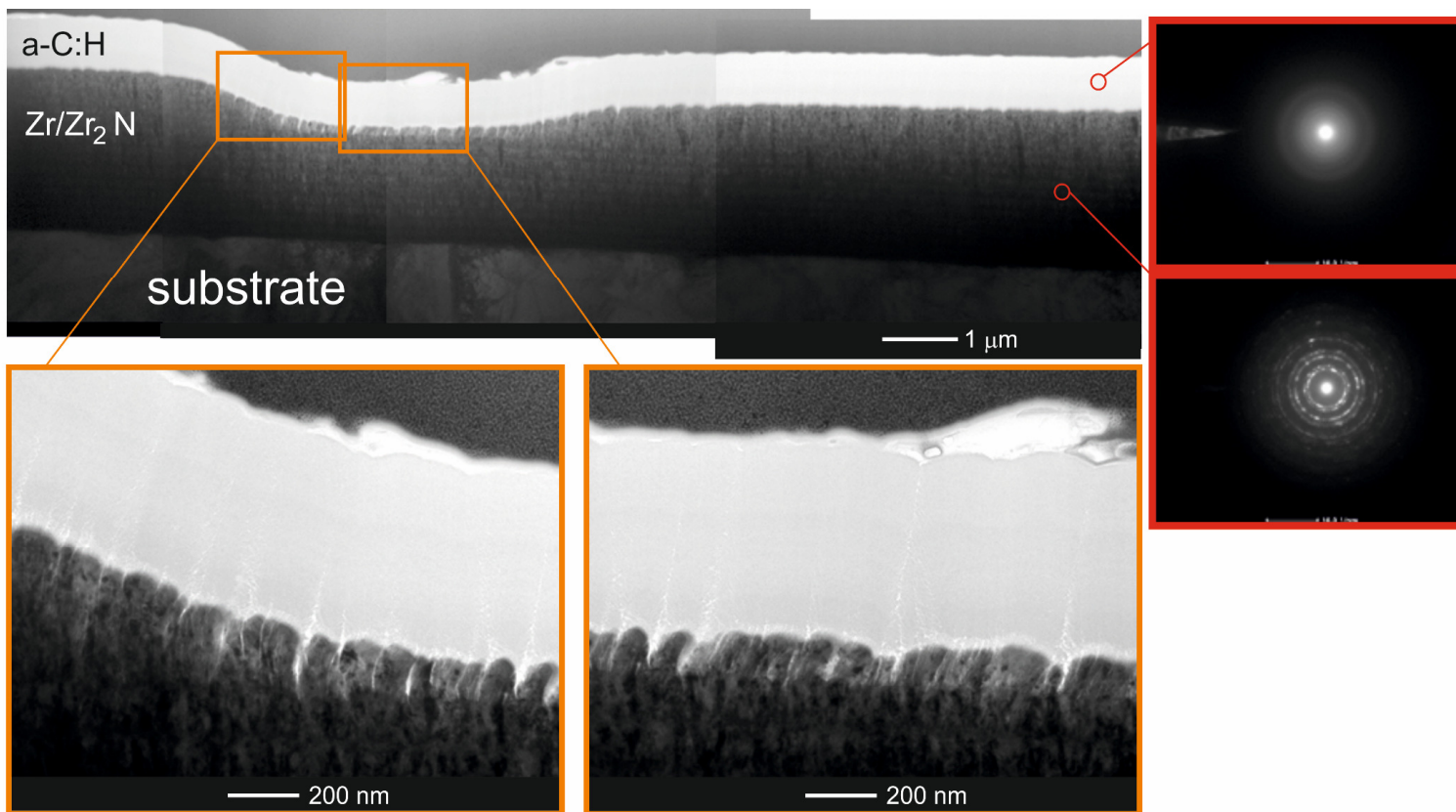
Presence of corrosive fluid components in metallic layers (increased sodium intensity)

SEM





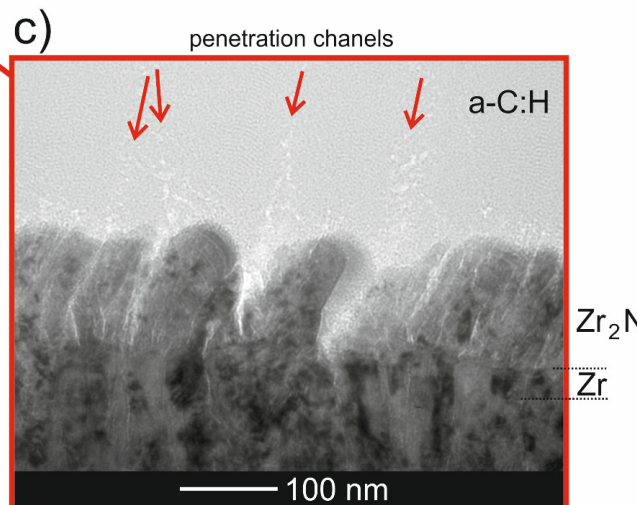
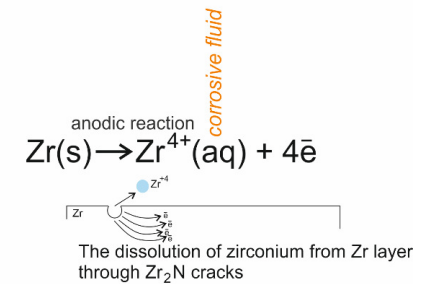
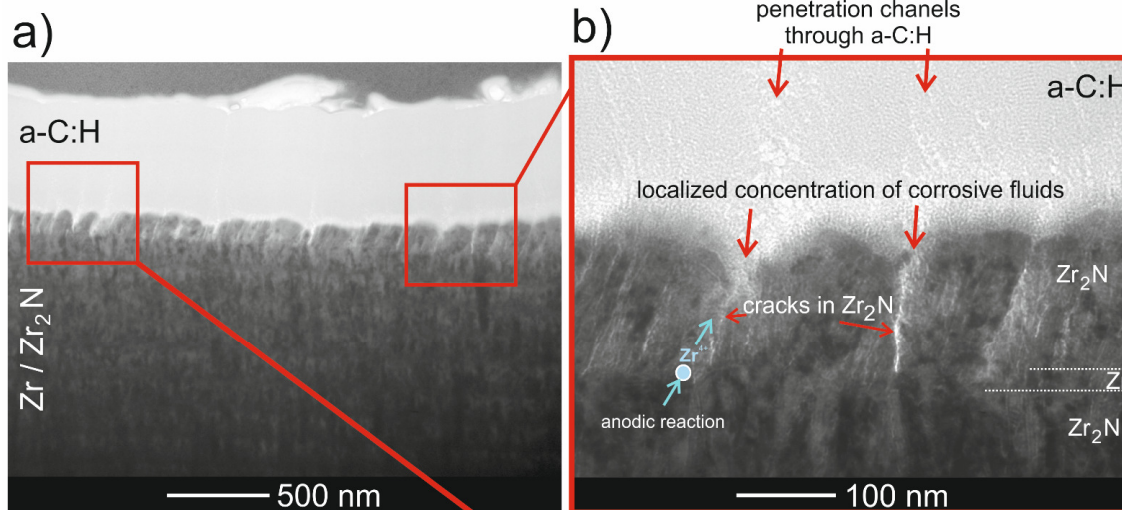
TEM BF + SAEDP



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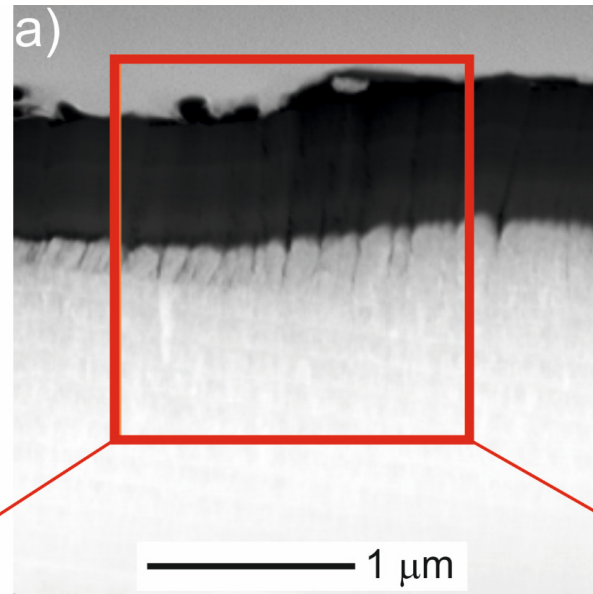
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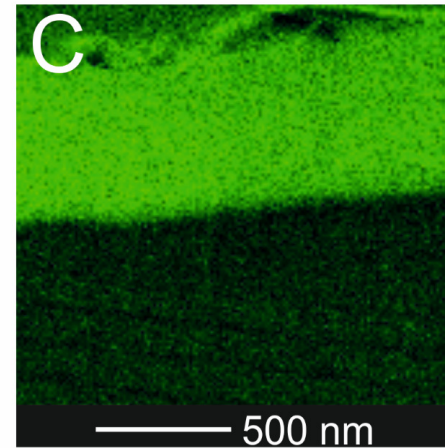
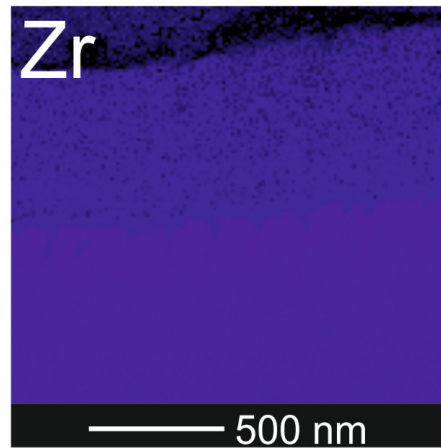
TEM BF

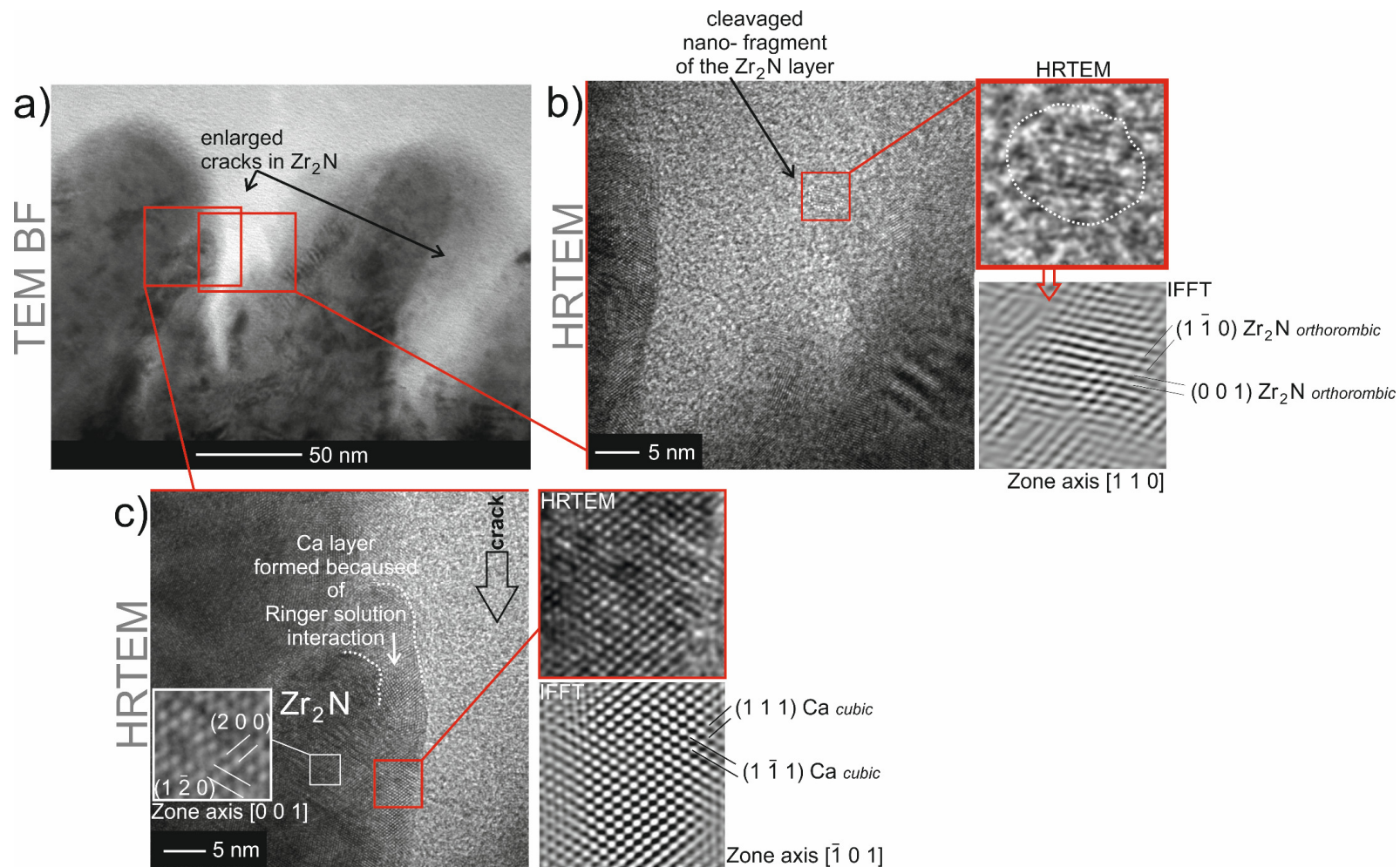


STEM + EDS



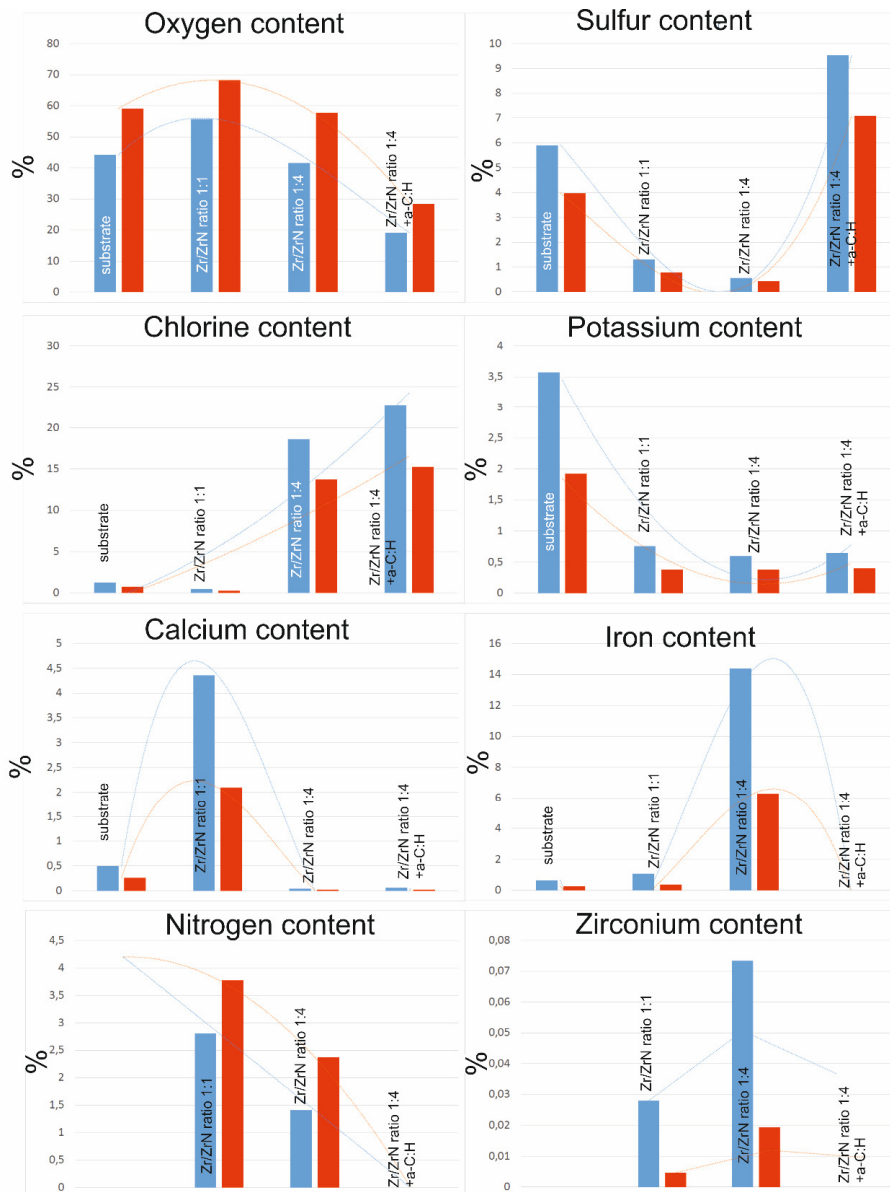
b)
zirconium content
in the a-C:H
structure
due to anodic reaction
in Zr layers





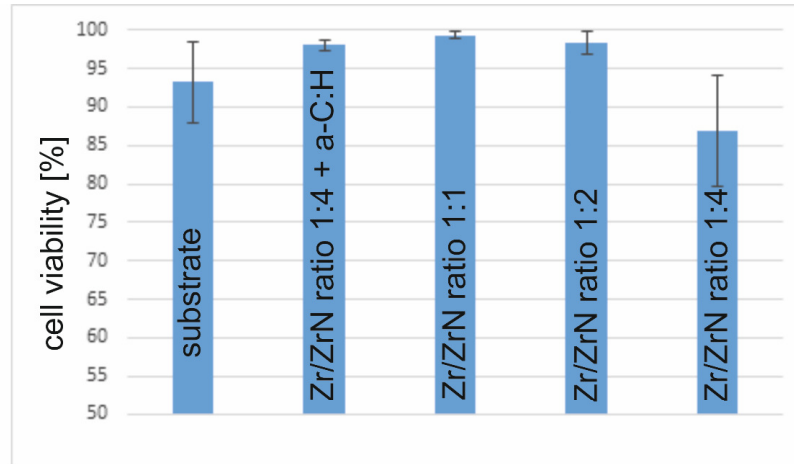


EDS

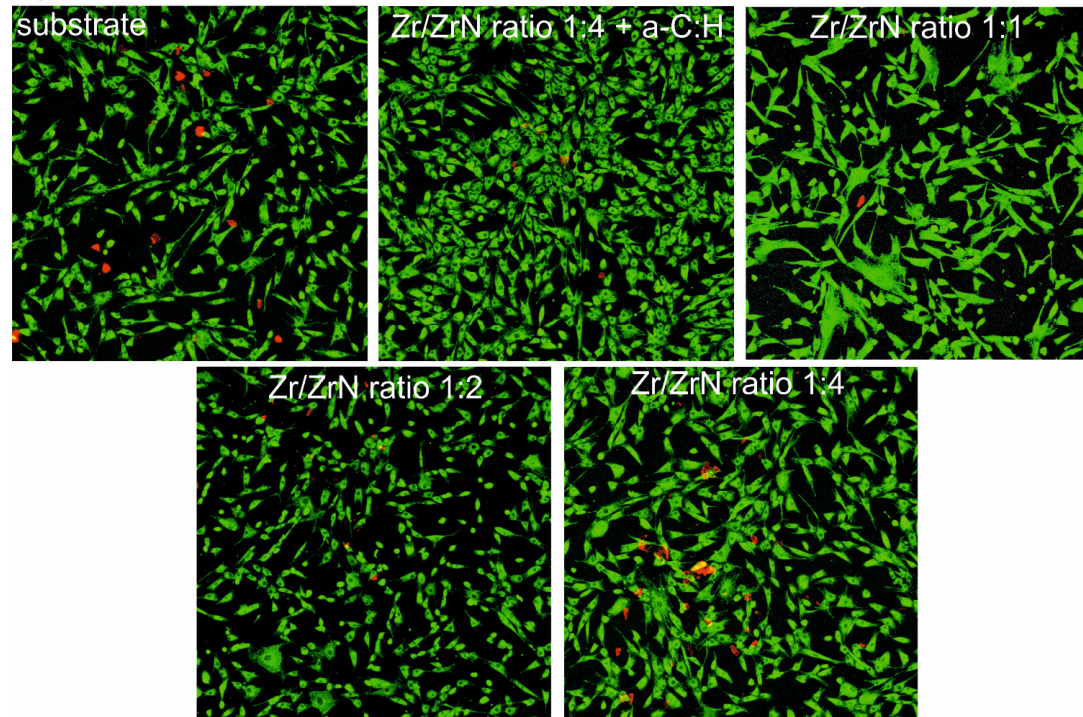




a)



b)





- Project NCN nr: 3066/B/T02/2011/40- *FINISHED*
- Project NCN nr: 2012/06/M/ST8/00408- *HARMONIA- FINISHED*
- Project NCN nr: 2012/07/B/ST8/03396- *OPUS- FINISHED*
- Project NCN nr: 2014/15/B/ST8/00103- *OPUS- FINISHED*
- Project NCN nr: 2015/19/B/ST8/00942- *OPUS- in progress*
- Project NCBR, number: DZP/M-ERA.NET-2015/285/2016- *in progress*



Title: Anti- bacterial optimization of high-strength, severe-plastic-deformed titanium alloys for spinal implants and surgical tools

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**FABRYKA NARZĘDZI
 MEDYCZYNYCH CHIRMED**
 Andrzej Misztela
 Mstowska 8
 PL-42-240 Rudniki
POLEN

UNSER ZEICHEN: PHA/LJU/WOB
VERSAND: persönliche Übergabe
 durch LJU
PROJEKT: MAT.05-15.GF.010-01
LIEFERSCHEIN: 2018031

Niklasdorf, 16. Februar 2018

Pos.	Menge	EH	Art.-Nr.	Artikelbezeichnung	Ihre Bestellung:
0010	6	Pieces	C260_3-3D <i>A</i> <i>11</i>	tweezers dummies (Nr.: 5 - matokiane) <i>nie mat</i> (Nr.: 6 - niematawane) <i>matowane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 5 atom% Ag coating thickness: 2,10 µm	according to the agreement with Dr. J. Lackner
0020	5	Pieces	C260_4-3D <i>B</i> <i>11</i>	tweezers dummies (Nr.: 5 - matokiane) - <i>nie matowane</i> (Nr.: 6 - niematawane) - <i>matowane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 3 atom% Ag coating thickness: 2,30 µm	
0030	4	Pieces	C260_5-3D <i>C</i> <i>11</i>	tweezers dummies (Nr.: 5 - matokiane) <i>nie matowane</i> (Nr.: 6 - niematawane) coated with <i>matowane</i> multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 7 atom% Ag coating thickness: 2,05 µm	



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Narodowe Centrum
Badań i Rozwoju



Rudniki k/Częstochowy dnia 15 marca 2018

Prof. Dr hab. inż. Roman Major
Instytut Metalurgii i Inżynierii Materiałowej
im. Aleksandra Krupkowskiego
Polskiej Akademii Nauk
ul. Reymonta 25
Kraków 30-059

W nawiązaniu do prowadzonej korespondencji pocztą elektroniczną w ramach projektu o akronimie SPD - bioTribio przekazuję do badań próbki w postaci pojedynczych bransz tytanowych pincet atraumatycznych z naniesionymi powłokami przez JR.

Próbki zostały poddane 18 krotnemu procesowi sterylizacji.

W załączeniu raport z ostatniego cyklu procesu sterylizacji oraz 6 szt. próbek o oznaczeniach:

STER-TEST/A5/1 i STER-TEST/A6/1, STER-TEST/B5/1 i STER-TEST/B6/1, STER-TEST/C5/1 i STER-TEST/C6/1

Znaczenie:

A5/1 - nr próbki C260_3-3D, powierzchnia narzędzia szcztokowana, sztuka pierwsza

A6/1 - nr próbki C260_3-3D, powierzchnia narzędzia matowana, sztuka pierwsza

B5/1 - nr próbki C260_4-3D, powierzchnia narzędzia szcztokowana, sztuka pierwsza

B6/1 - nr próbki C260_4-3D, powierzchnia narzędzia matowana, sztuka pierwsza

C5/1 - nr próbki C260_5-3D, powierzchnia narzędzia szcztokowana, sztuka pierwsza

C6/1 - nr próbki C260_5-3D, powierzchnia narzędzia matowana, sztuka pierwsza

Wszelkie informacje proszę o przekazywanie na poniższy kontakt:

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Regon 150376074, NIP 949-148-89-57
fax 034 320-15-00, tel. 034 320-14-84

DYREKTOR

Inż. Marek Dyner



European Union
European Social Fund





European

INSTITUTE OF METALURGY

European Union



HL0VK722

MELAG Vacuklav 24-B/L

Program : Program szybki B
 134°C opakowane
 Data : 13.03.2018
 Czas : 07:32:32 (Start)
 Nr. cyklu : 2
 SN : 201724-BL1136

Podgrzewanie 109.4 °C
 AIN6: Przewodność 0 µS/cm

Postep programu	Cisn. bar	Temperat. °C	Czas min
Start	0.00	64.8	00:00
1. Frakcjonowanie			
Ewakuacja	-0.92	59.9	01:15
Wlot pary	0.41	107.6	03:45
2. Frakcjonowanie			
Ewakuacja	-0.82	59.3	05:22
Wlot pary	0.40	108.9	07:14
3. Frakcjonowanie			
Ewakuacja	-0.82	59.7	08:54
Wlot pary	0.40	108.9	10:44
Wzrost cisn.	2.05	134.0	14:20
Steryln.początek	2.05	134.0	14:20
Steryln.koniec	2.19	135.6	19:50
Upust ciśnienia	0.18	105.4	20:32
Suszenie próżniowe			
Suszenie pocz.	-0.31	91.7	20:46
Suszenie cisn.	-0.92	77.2	22:44
Suszenie cisn.	-0.94	86.2	24:44
Suszenie cisn.	-0.94	86.9	26:44
Suszenie cisn.	-0.94	85.8	28:44
Suszenie cisn.	-0.94	85.1	30:44
Suszenie cisn.	-0.94	84.9	32:44
Suszenie koniec	-0.90	85.0	32:46
Napowietrzanie	-0.28	86.1	33:04
Koniec	0.00	86.6	33:21

PROGRAM PRAWIDŁOWO ZAKOŃCZONY!

Temperatura : 135.4 +0.3 /-0.2 °C
 Ciśnienie : 2.17 +0.02/-0.02 bar
 Czas steryliz. : 5 min 30 s
 Czas : 08:05:53 (Koniec)

Strona 1

HL0VK722

77 201701136 5.15 5.06
 CRC: 0x297D MF V2.008A

Program : Program szybki B
 134°C opakowane
 Data : 13.03.2018
 Czas : 07:32:32 (Start)
 Nr. cyklu : 2
 SN : 201724-BL1136

Podgrzewanie 109.4 °C
 AIN6: Przewodność 0 µS/cm

Postep programu	Cisn. bar	Temperat. °C	Czas min
Start	0.00	64.8	00:00
1. Frakcjonowanie			
Ewakuacja	-0.92	59.9	01:15
Wlot pary	0.41	107.6	03:45
2. Frakcjonowanie			
Ewakuacja	-0.82	59.3	05:22
Wlot pary	0.40	108.9	07:14
3. Frakcjonowanie			
Ewakuacja	-0.82	59.7	08:54
Wlot pary	0.40	108.9	10:44
Wzrost cisn.	2.05	134.0	14:20
Steryln.początek	2.05	134.0	14:20
Steryln.koniec	2.19	135.6	19:50
Upust ciśnienia	0.18	105.4	20:32
Suszenie próżniowe			
Suszenie pocz.	-0.31	91.7	20:46
Suszenie cisn.	-0.92	77.2	22:44
Suszenie cisn.	-0.94	86.2	24:44
Suszenie cisn.	-0.94	86.9	26:44
Suszenie cisn.	-0.94	85.8	28:44
Suszenie cisn.	-0.94	85.1	30:44
Suszenie cisn.	-0.94	84.9	32:44
Suszenie koniec	-0.90	85.0	32:46
Napowietrzanie	-0.28	86.1	33:04
Koniec	0.00	86.6	33:21

PROGRAM PRAWIDŁOWO ZAKOŃCZONY!

Temperatura : 135.4 +0.3 /-0.2 °C
 Ciśnienie : 2.17 +0.02/-0.02 bar
 Czas steryliz. : 5 min 30 s
 Czas : 08:05:53 (Koniec)

Strona 2



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 POLEN

UNSER ZEICHEN: PHA/LJU/WOB
 VERSAND: persönliche Übergabe
 durch LJU
 PROJEKT: MAT.05-15.GF.010-01
 LIEFERSCHEIN: 2018031

Niklasdorf, 16. Februar 2018

Pos.	Menge	EH	Art.-Nr.	Artikelbezeichnung	Ihre Bestellung:
0010	6	Pieces	C260_3-3D	tweezers dummies (Nr.: 5 - matokiane) <i>nie matokiane</i> (Nr.: 6 - niematawane) <i>matokiane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 5 atom% Ag coating thickness: 2,10 µm	according to the agreement with DDr. J. Lackner
0020	5	Pieces	C260_4-3D	tweezers dummies (Nr.: 5 - matokiane) <i>- nie matokiane</i> (Nr.: 6 - niematawane) <i>- matokiane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 3 atom% Ag coating thickness: 2,30 µm	
0030	4	Pieces	C260_5-3D	tweezers dummies (Nr.: 5 - matokiane) <i>nie matokiane</i> (Nr.: 6 - niematawane) <i>matokiane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 7 atom% Ag coating thickness: 2,05 µm	



C260_3_3D

A

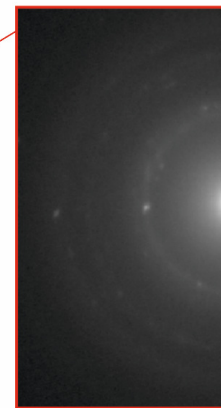
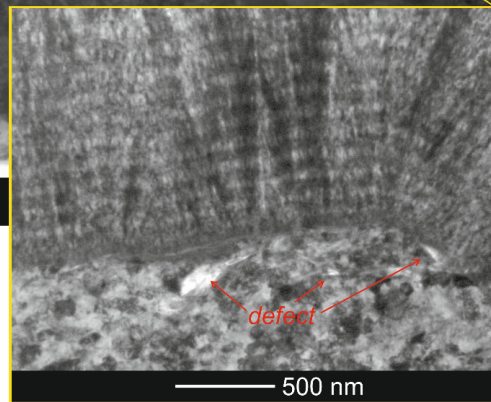
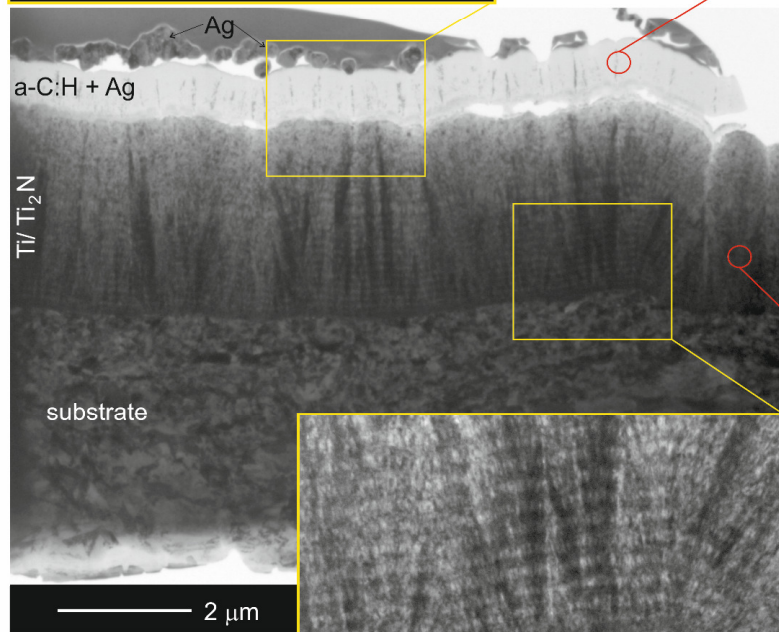
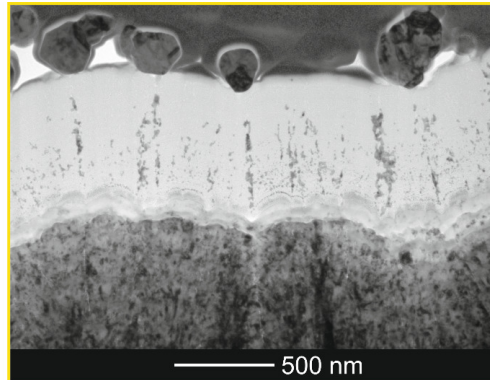
Project WND-POWR.03.02.00-00-1043/16

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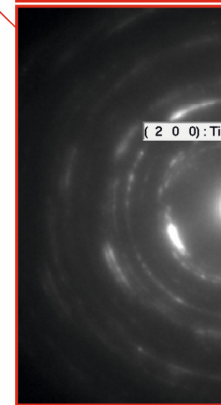
Project co-financed by the European Union within the European Social Funds



TEM BF + SAEDP



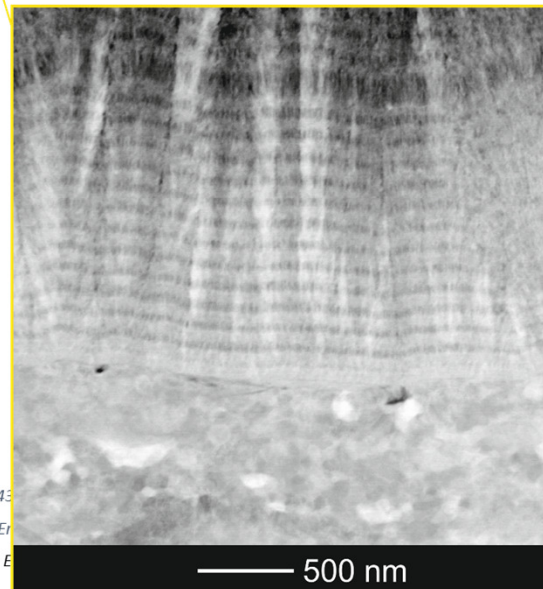
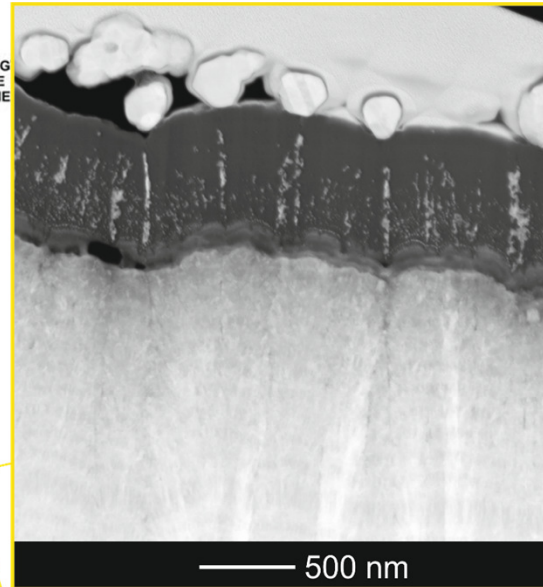
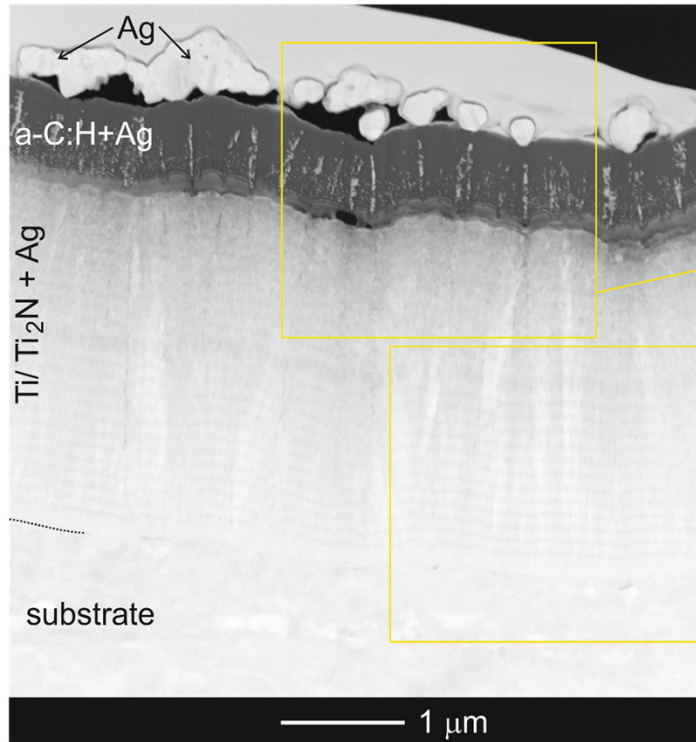
(1 1 1): Ag
(2 2 0): Ag
(3 1 1): Ag



(4 2 2): TiN
(2 2 2): TiN
(2 2 0): TiN
(2 0 0): TiN
(1 1 1): Ti (1 1 0): Ti
(1 1 2): Ti2N (3 0 3): Ti2N
(2 2 4): Ti2N
(3 3 1): Ag

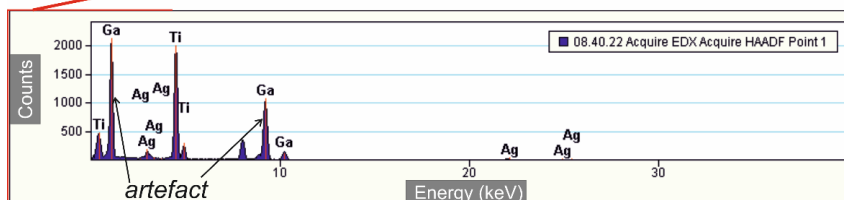
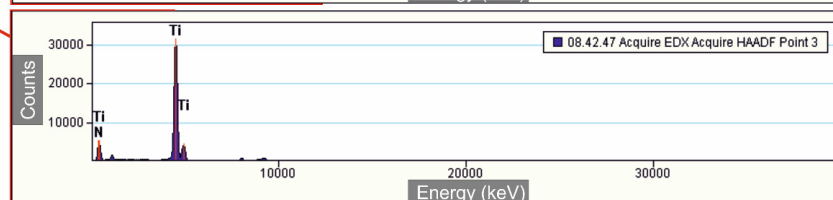
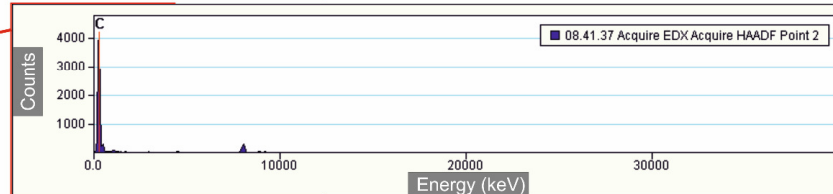
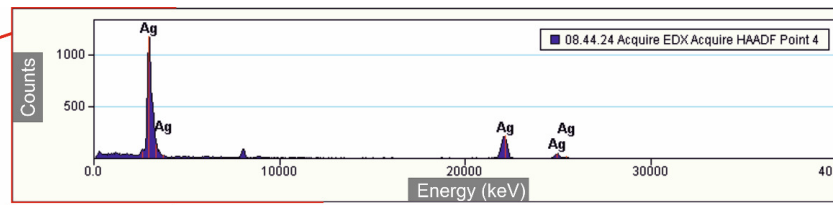
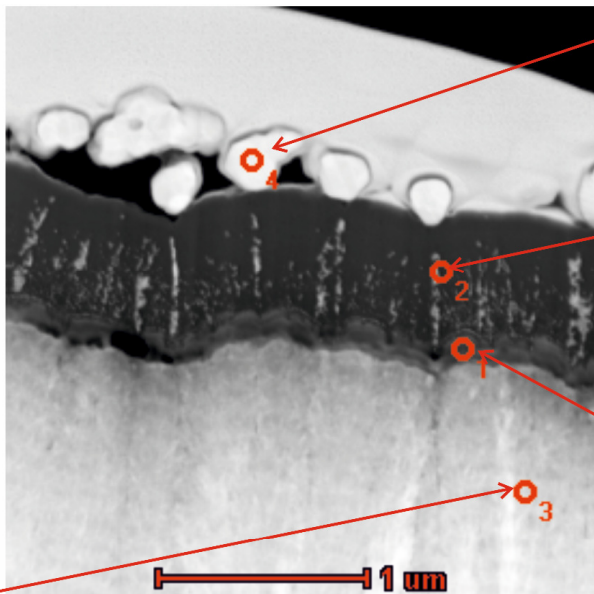


STEM



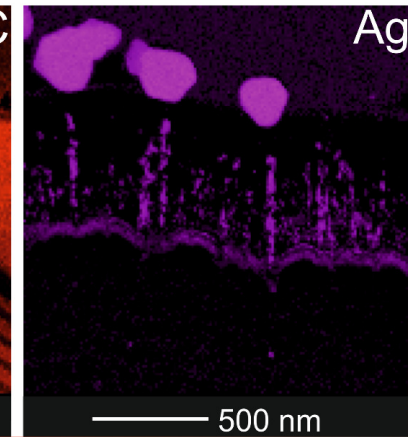
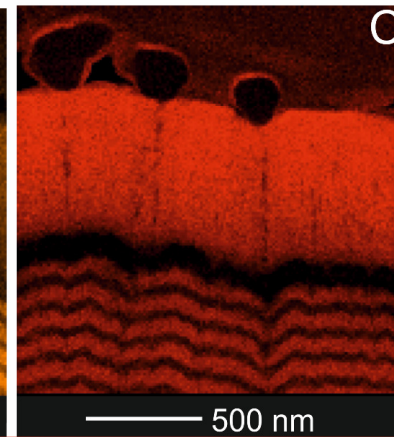
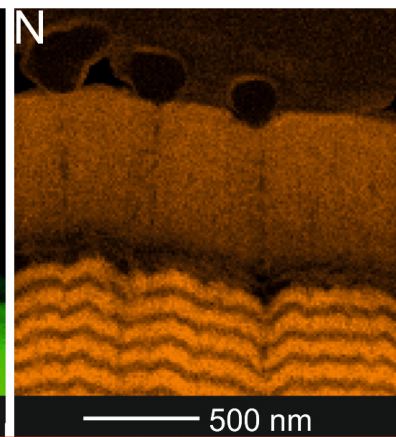
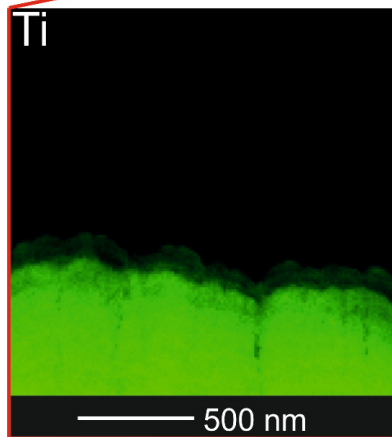
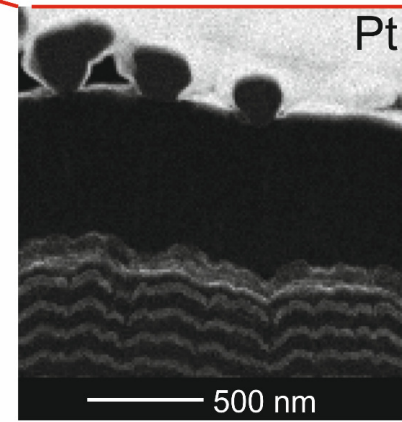
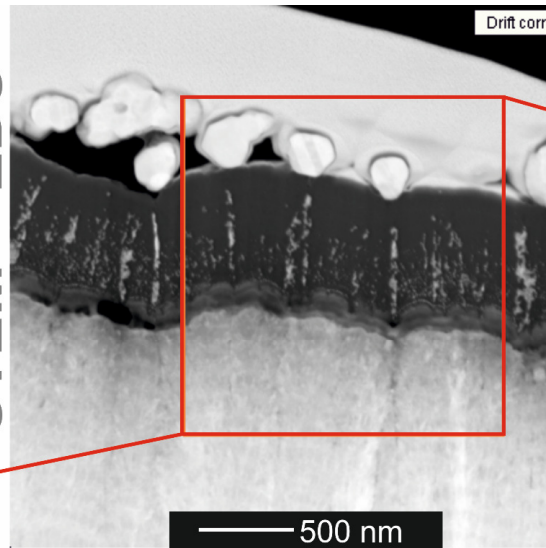


STEM + EDS

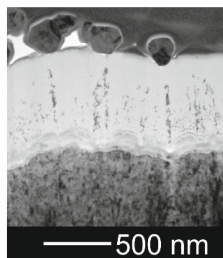




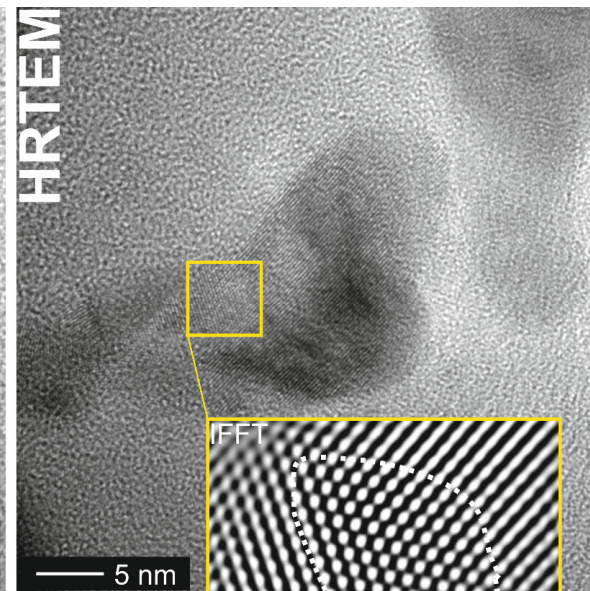
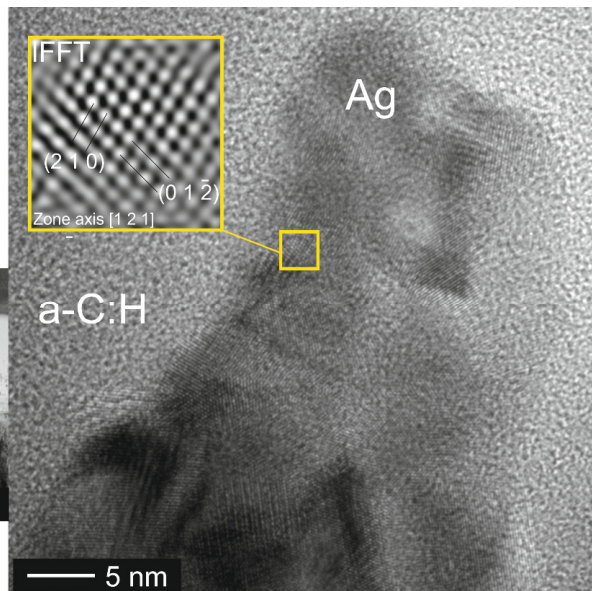
STEM + EDS



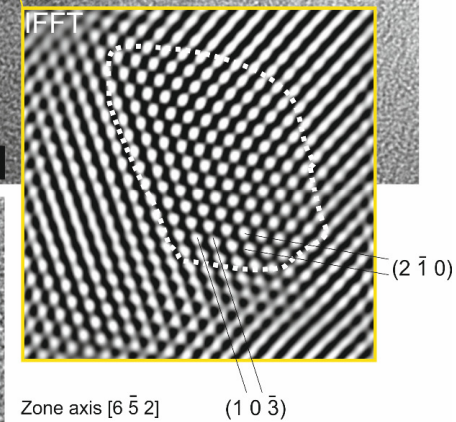
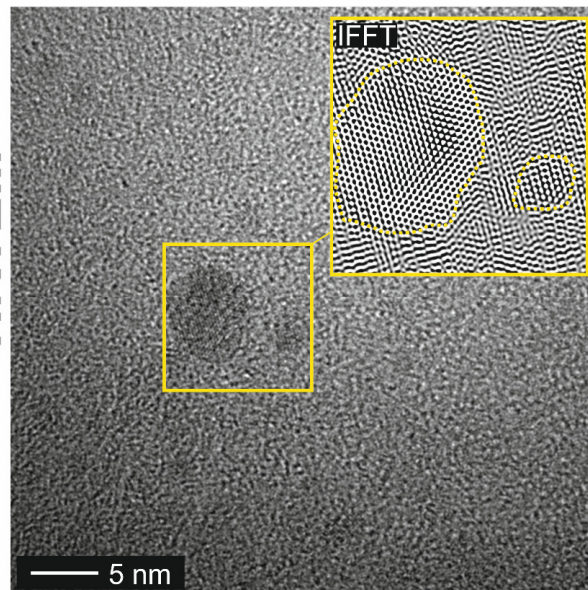
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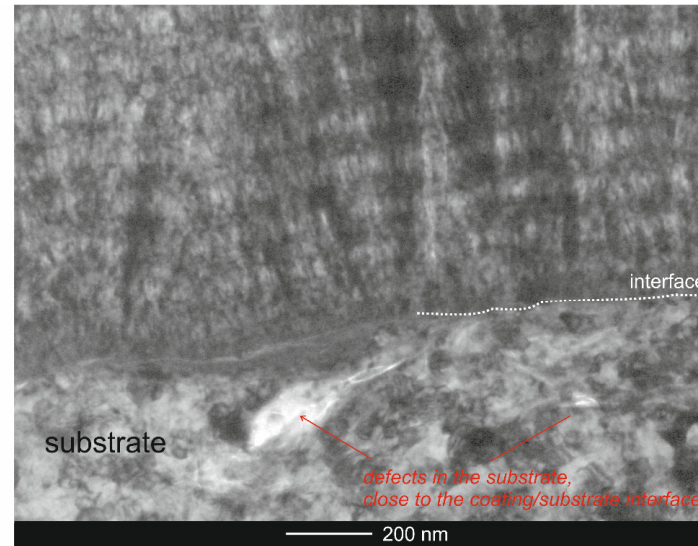
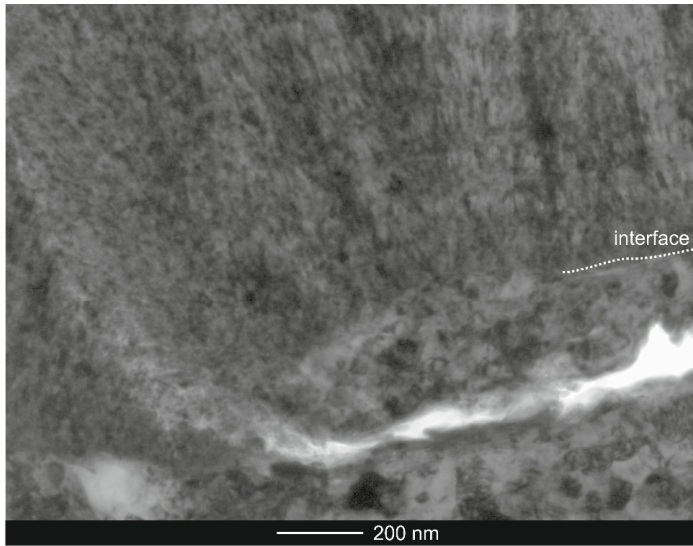
HRTEM



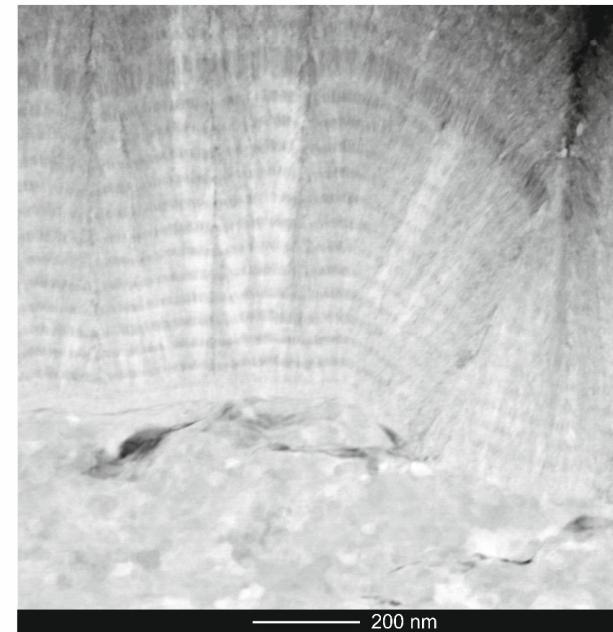
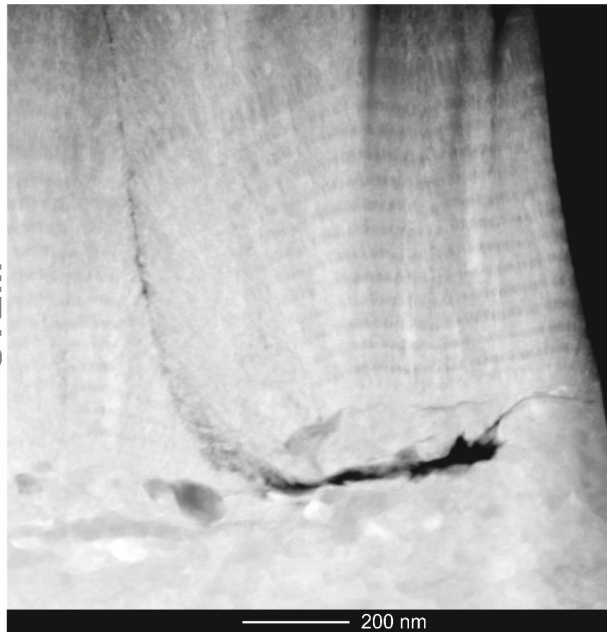
HRTEM



TEM BF



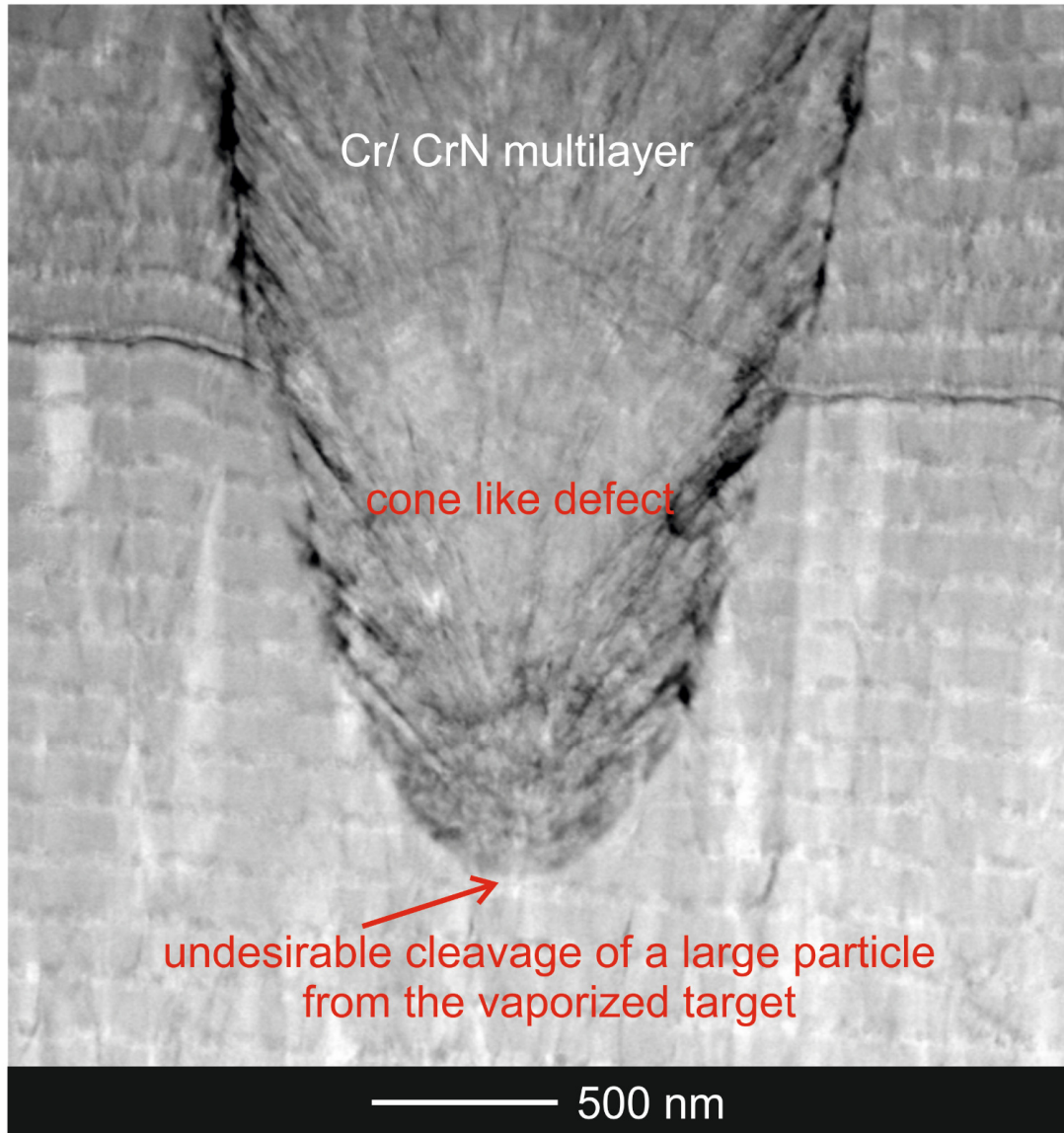
STEM



EXAMPLE FROM OUR PREVIOUS RESEARCH



STEM





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 Andrzej Misztela
 Mstowska 8
 PL-42-240 Rudniki
 POLEN

UNSER ZEICHEN: PHA/LJU/WOB
 VERSAND: persönliche Übergabe
 durch LJU
 PROJEKT: MAT.05-15.GF.010-01
 LIEFERSCHEIN: 2018031

Niklasdorf, 16. Februar 2018

Pos.	Menge	EH	Art.-Nr.	Artikelbezeichnung	Ihre Bestellung:
0010	6	Pieces	C260_3-3D	<i>A</i> tweezers dummies (Nr.: 5 - matokiane) <i>nie matokiane</i> (Nr.: 6 - niematawane) <i>matokiane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 5 atom% Ag coating thickness: 2,10 µm	according to the agreement with Dr. J. Lackner
0020	5	Pieces	C260_4-3D	<i>B</i> tweezers dummies (Nr.: 5 - matokiane) <i>nie matokiane</i> (Nr.: 6 - niematawane) <i>matokiane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 3 atom% Ag	
0030	4	Pieces	C260_5-3D	<i>C</i> tweezers dummies (Nr.: 5 - matokiane) <i>nie matokiane</i> (Nr.: 6 - niematawane) <i>matokiane</i> coated with multilayer 24x (Ti / TiN) = 1:1 doped 7,5 atom% Ag + a-C:H doped with 7 atom% Ag coating thickness: 2,05 µm	



C260_5_3D

C

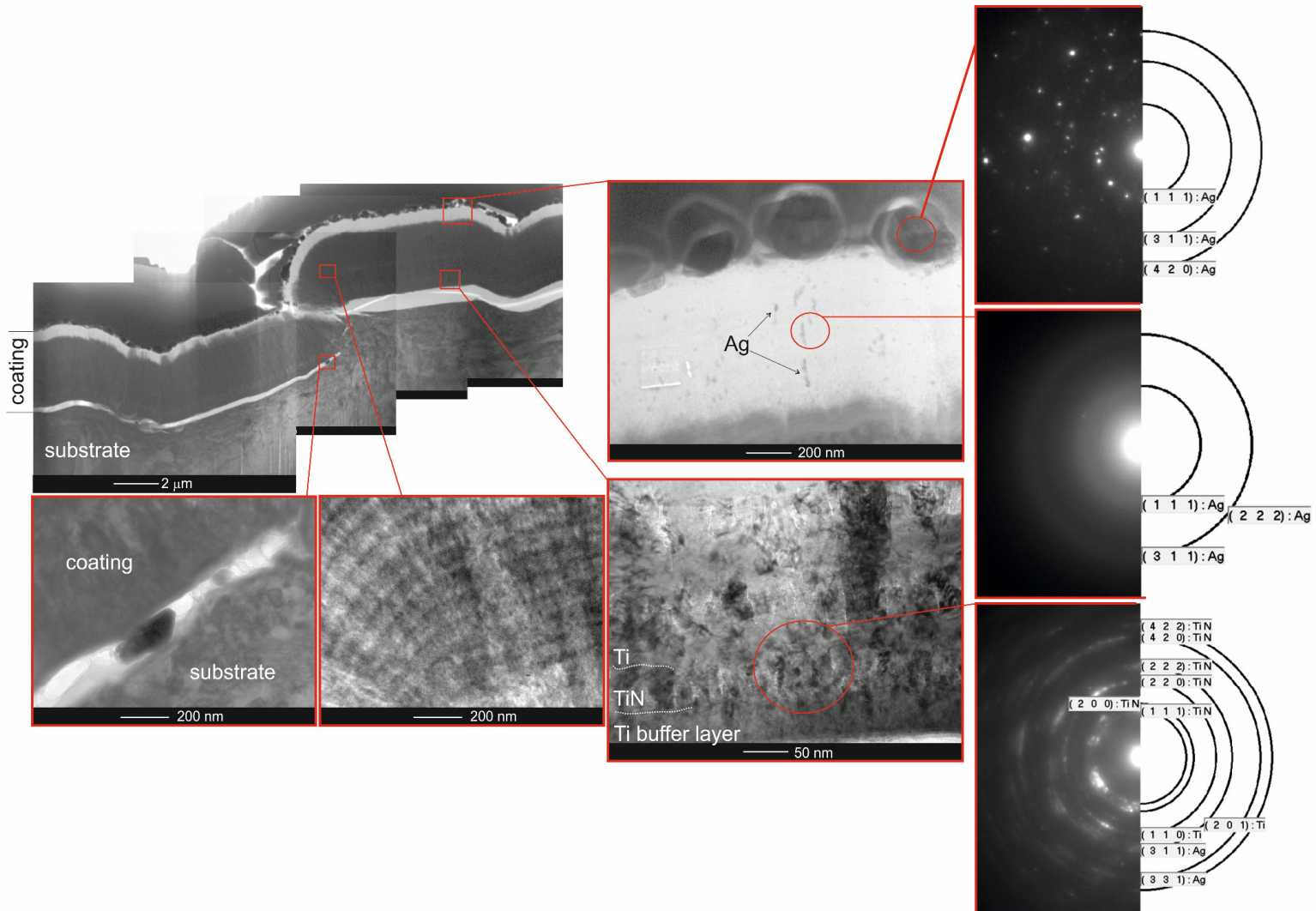
Project WND-POWR.03.02.00-00-1043/16

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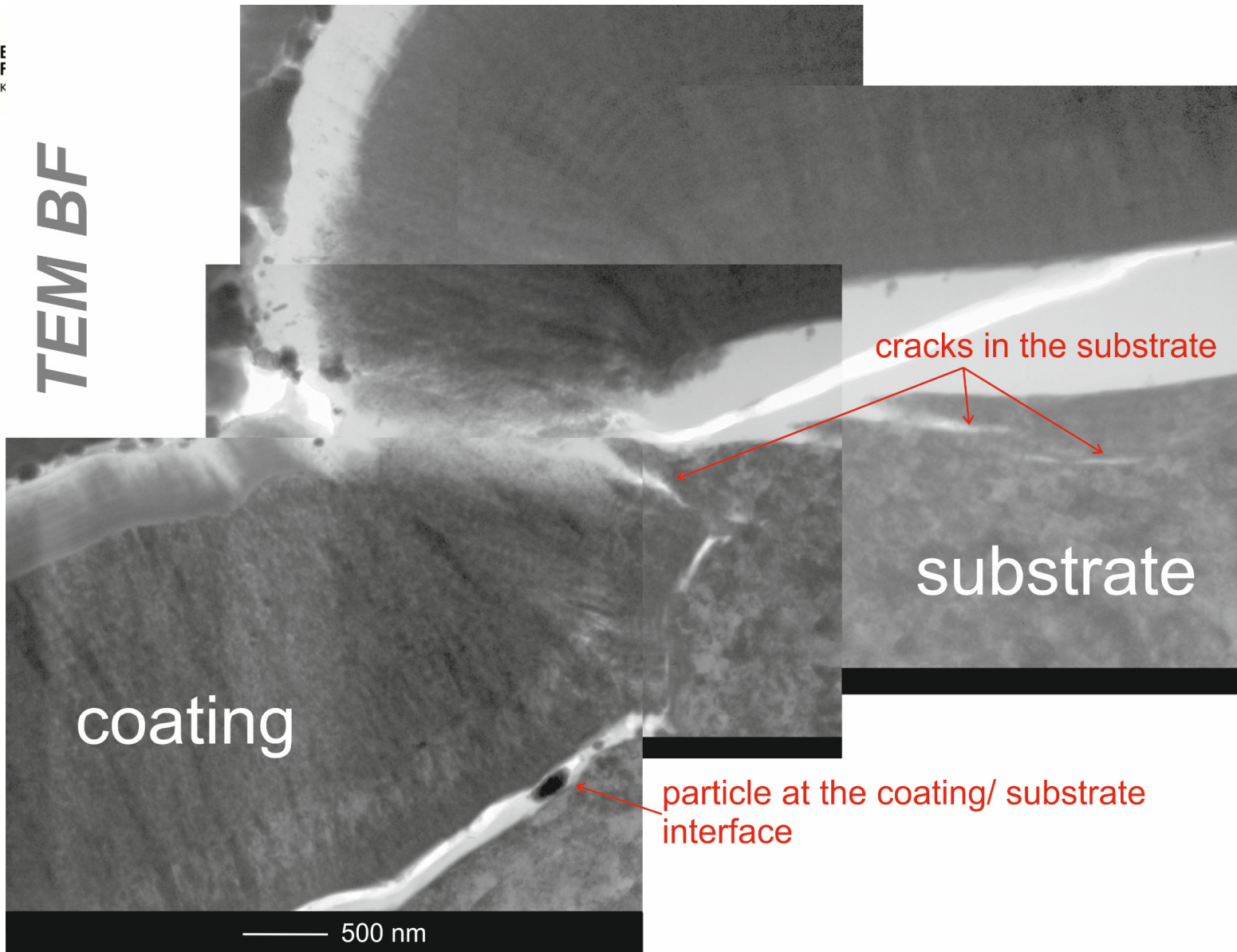


TEM BF + SAEDP



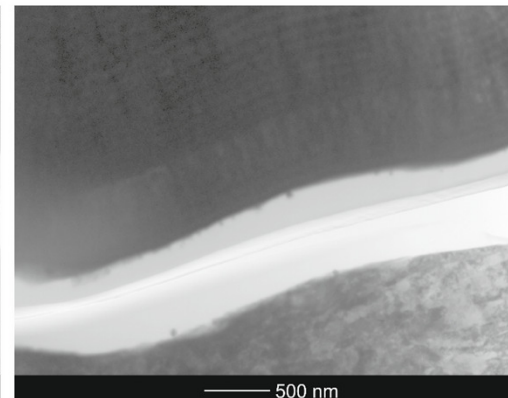
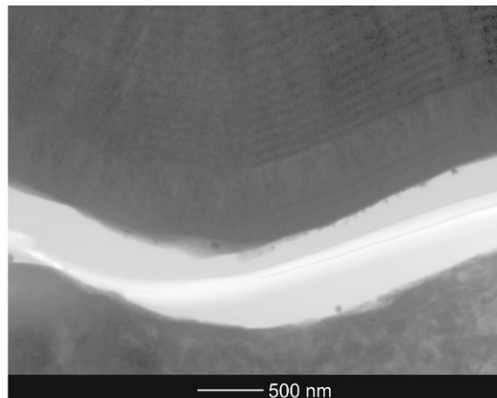
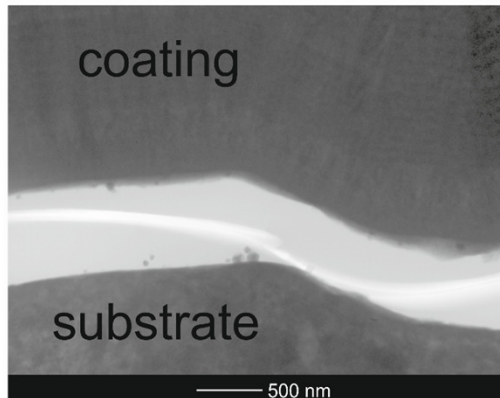


TEM BF





TEM BF



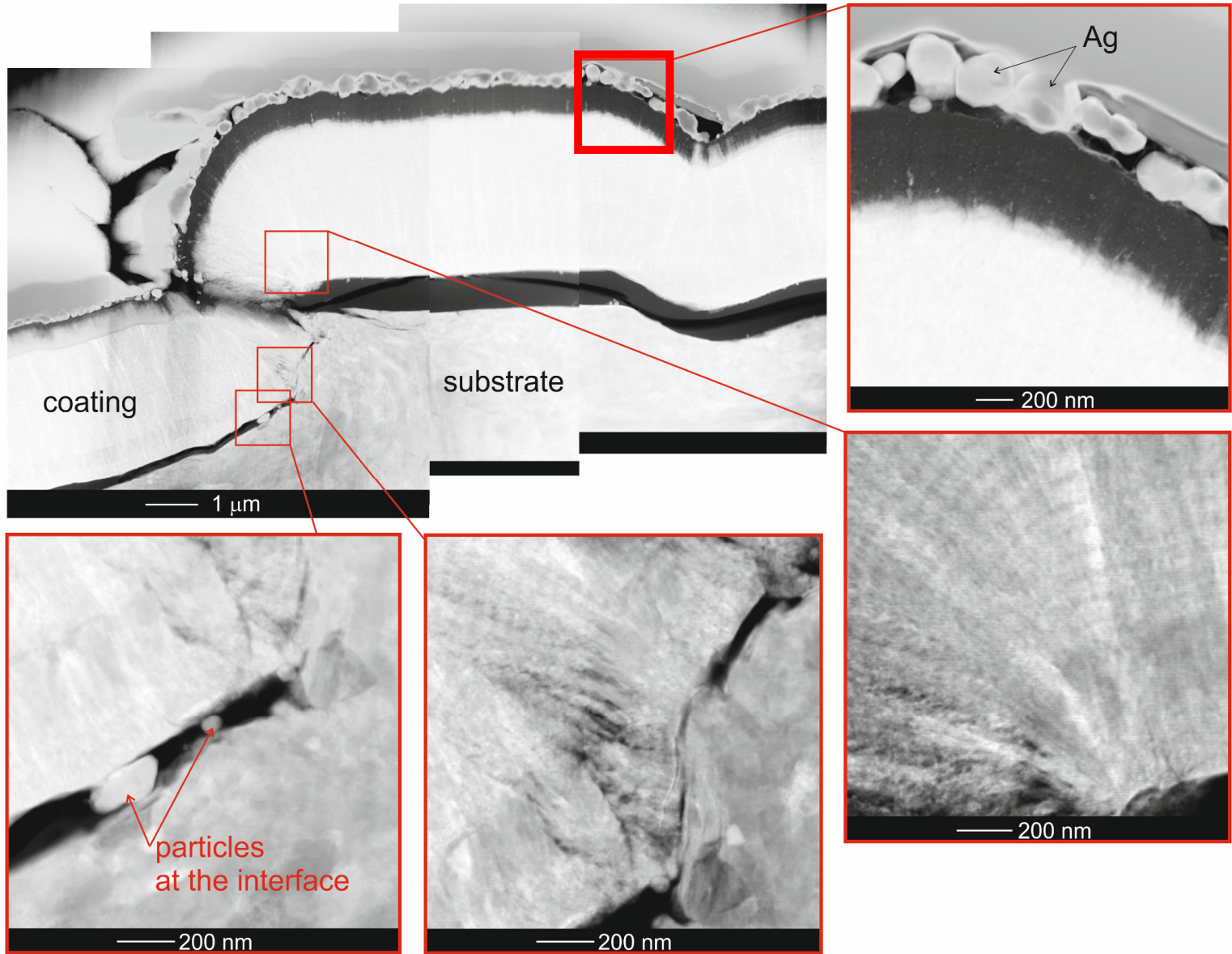
Project WND-POWR.03.02.00-00-1043/16

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STEM



coating

substrate

Ag

1 μm

200 nm

particles at the interface

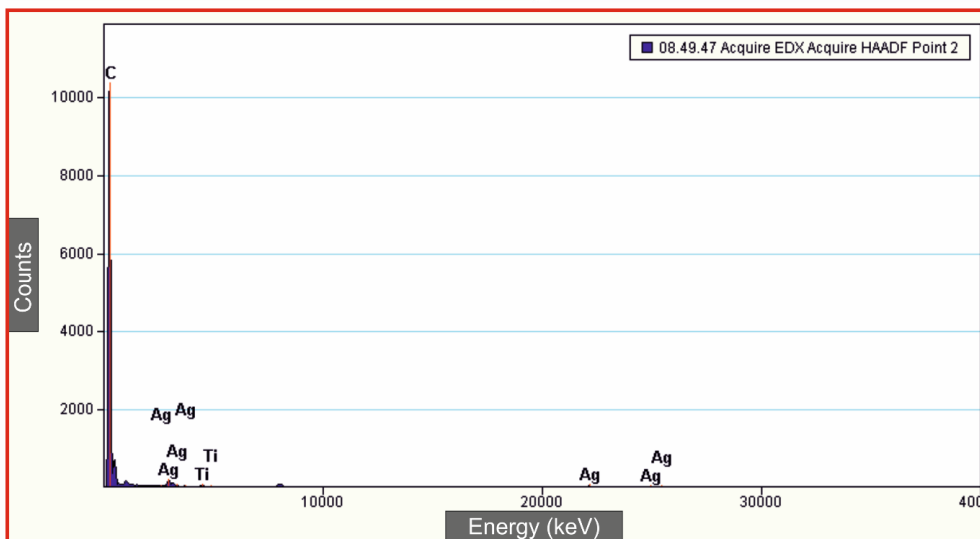
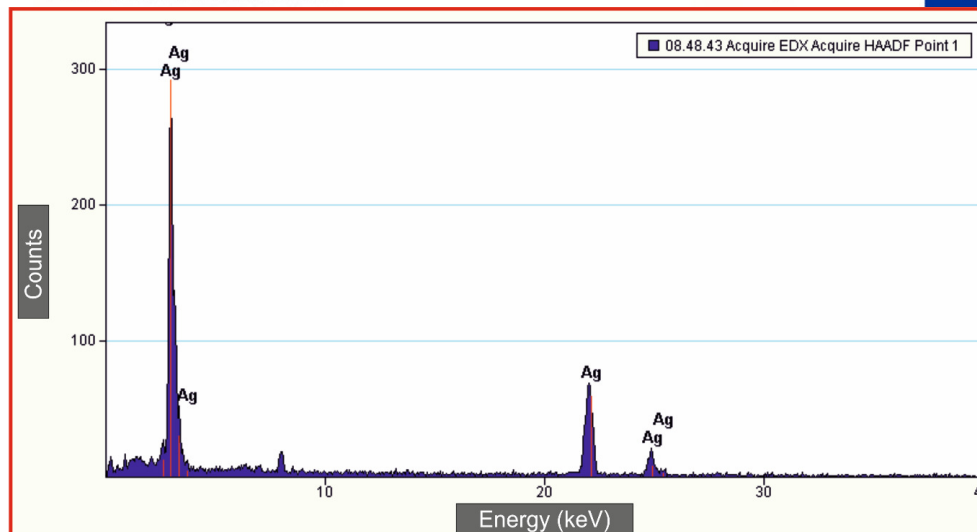
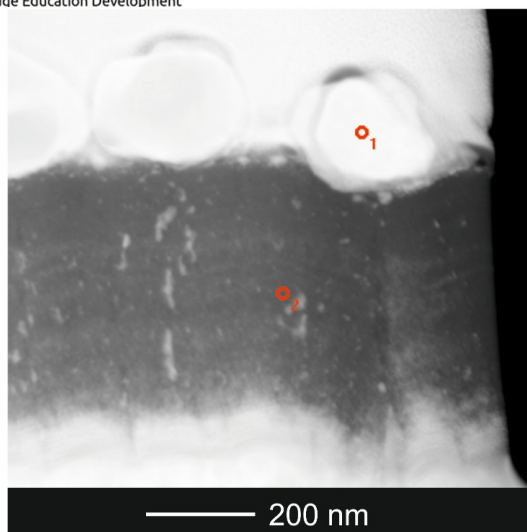
200 nm

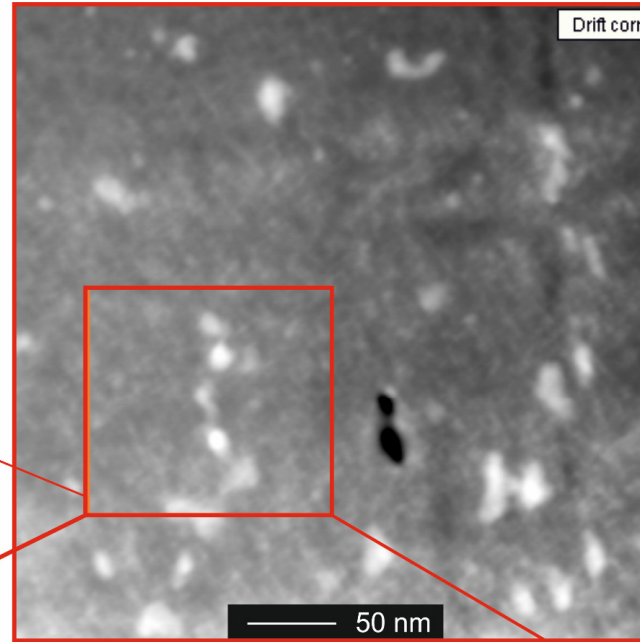
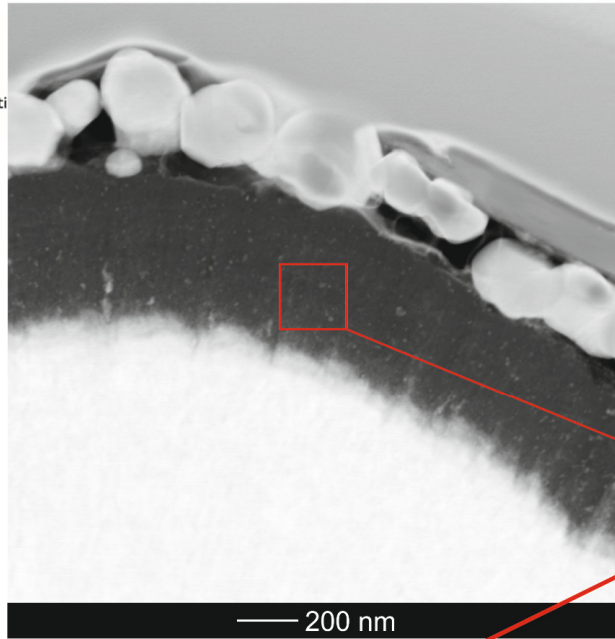
200 nm

200 nm

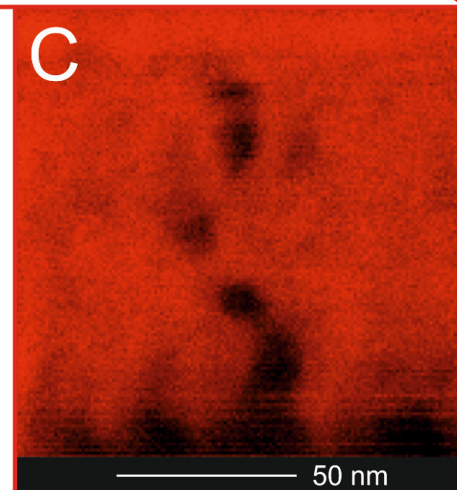
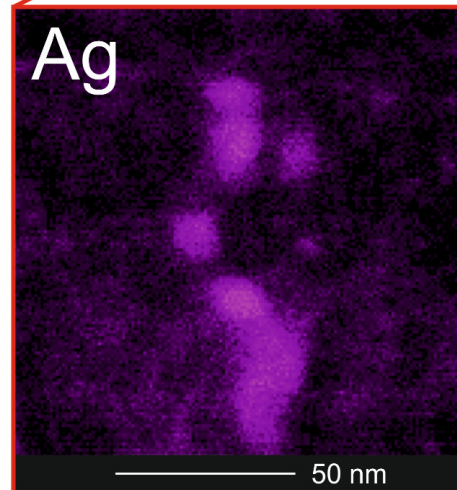


STEM + EDS



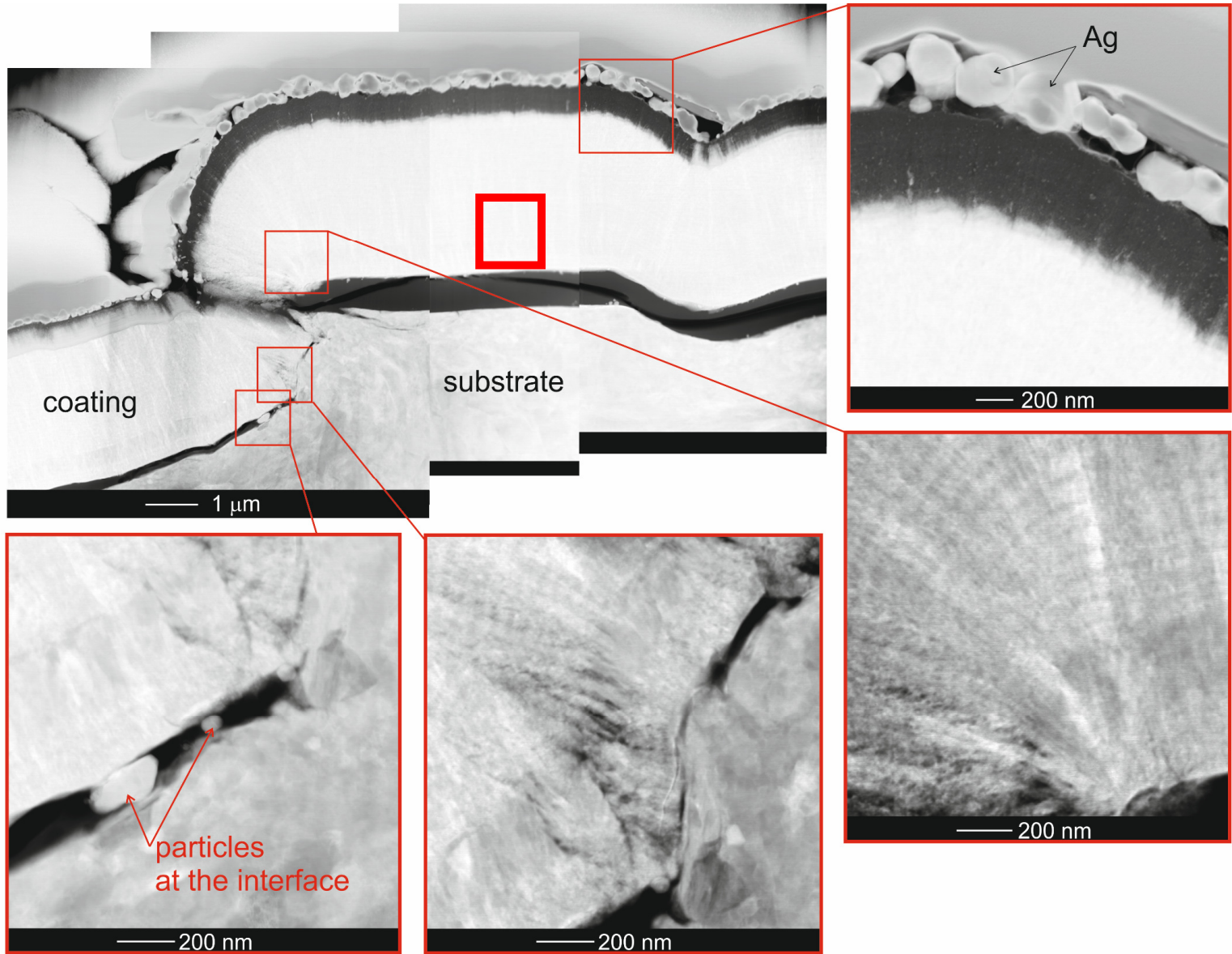


STEM + EDS

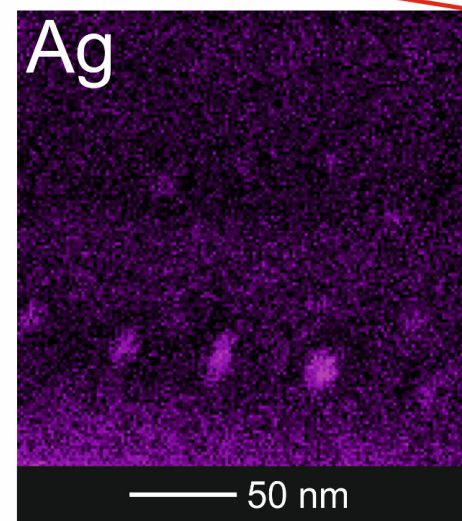
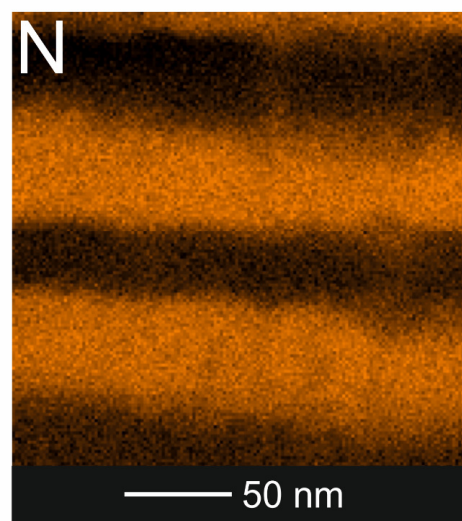
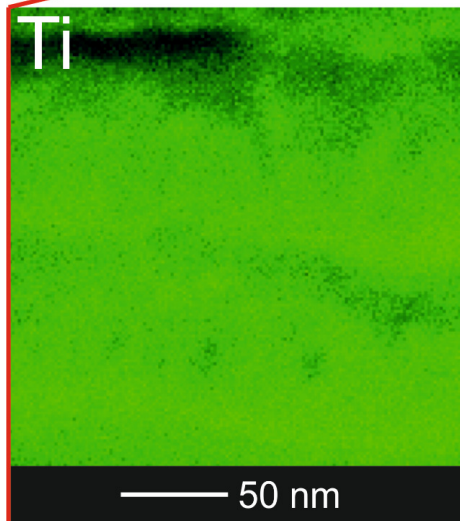
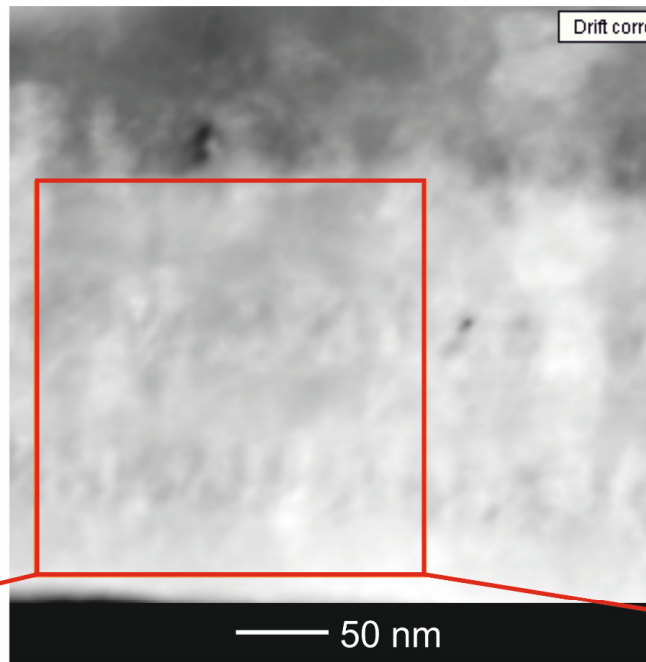




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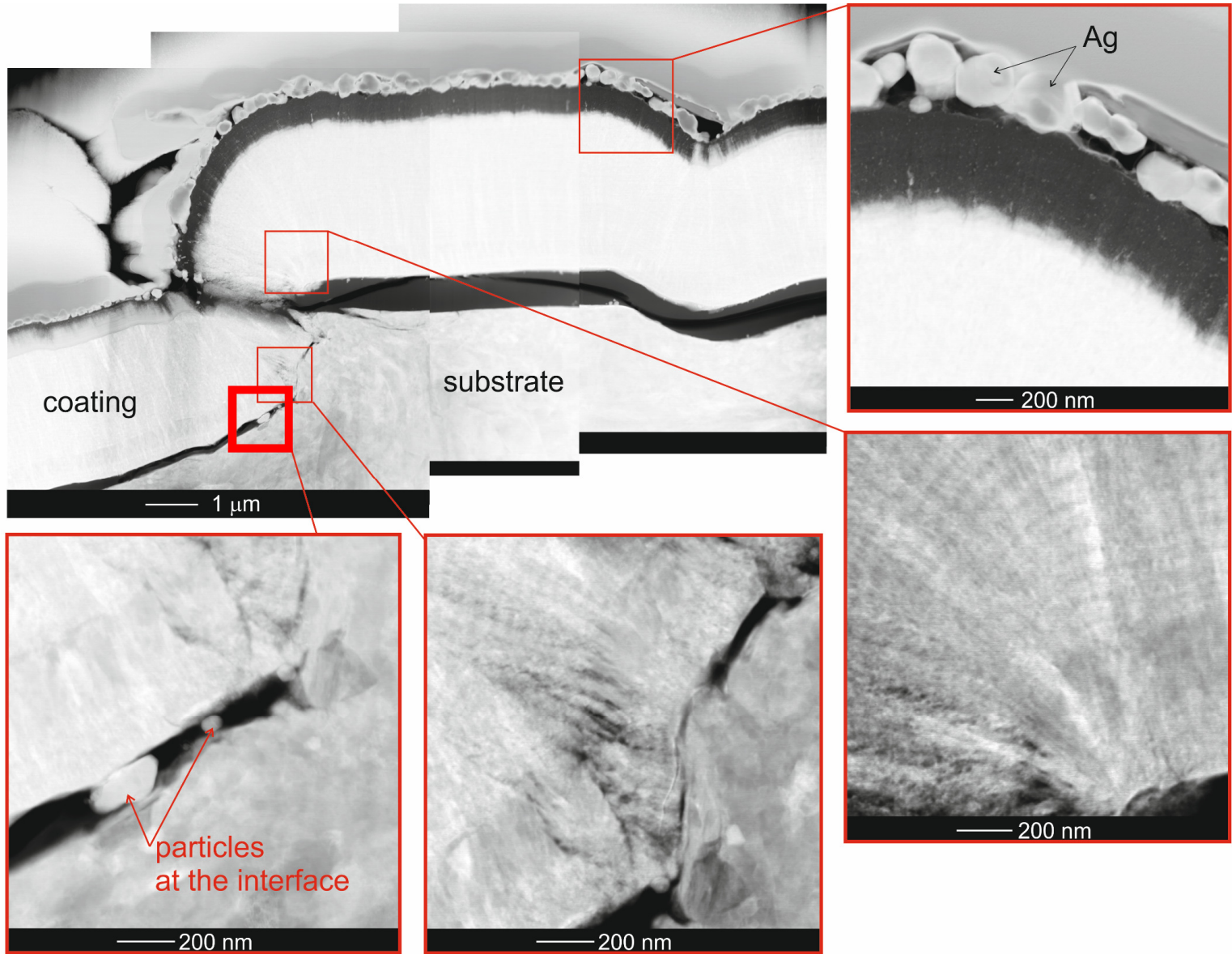


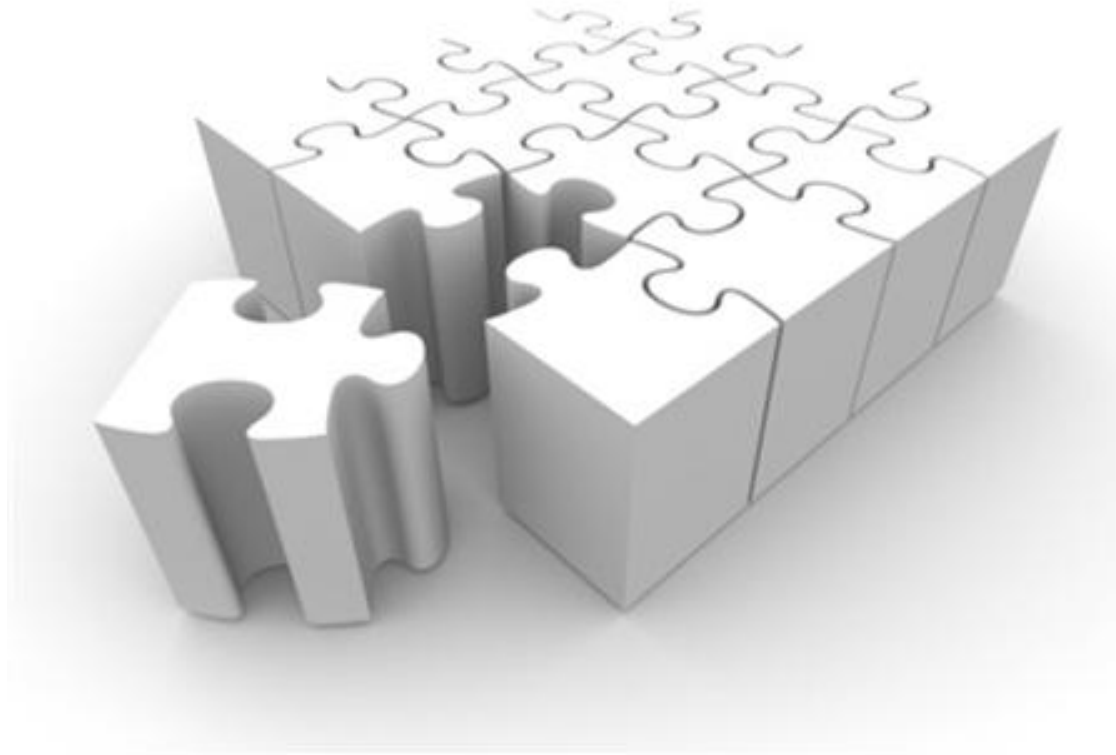
STEM + EDS





STEM





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