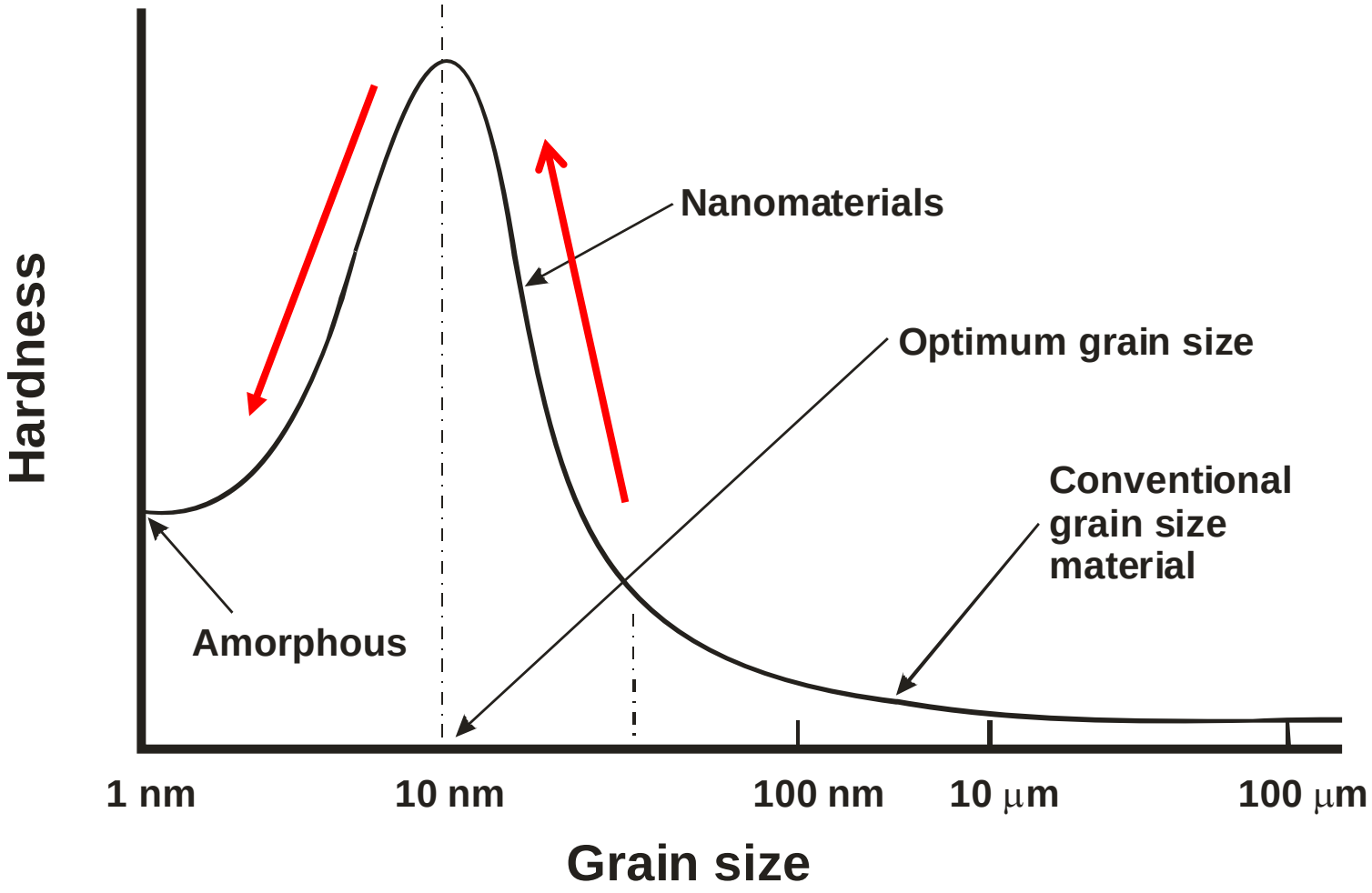
Silesian University of Technology

Microstructure and mechanical properties of duplex (Me)N/DLC coatings produced by lateral rotating cathode arc PVD and PACVD process

Krzysztof Lukaszkowicz

The purpose of this work is to examine the microstructure, mechanical and tribological properties of duplex nanocrystalline nitride (CrAlSiN, AlTiCrN, CrN) and amorphous topcoat DLC coatings produced by lateral rotating cathode arc PVD and PACVD technique respectively on the X40CrMoV5-1 hot work tool steel substrate.

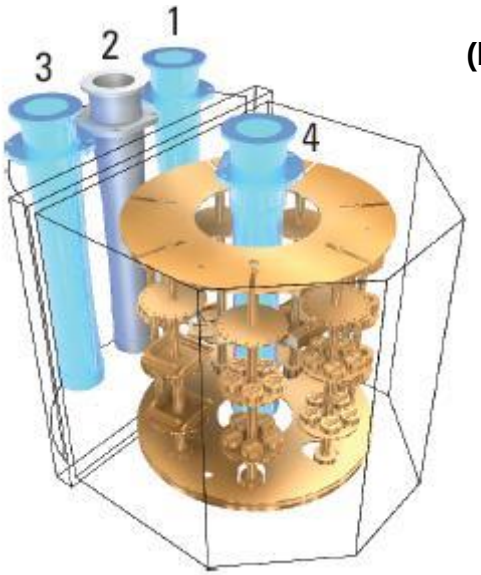


Hardness of material as a function of grain size

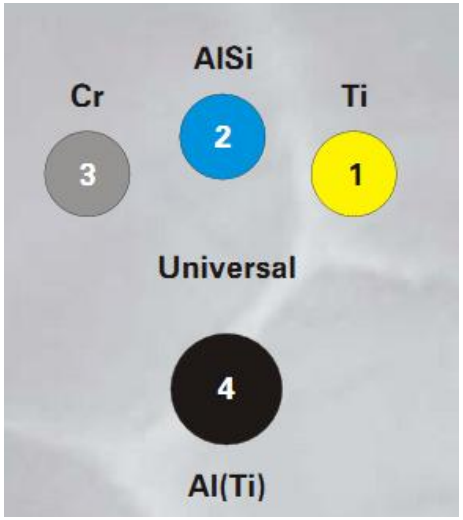
Materials

- **Substrate:**
  - ✓ hot work tool steel X40CrMoV5-1;
- **Coatings:**
  - ✓ CrAlSiN+DLC
  - ✓ AlTiCrN+DLC
  - ✓ CrN+DLC

(a)



(b)



PLATIT Pi300 industrial compact coating unit based on ARC PVD and PACVD technology: a) front view, b) typical cathode configuration

Deposition parameters

Parameters	CrAlSiN	AlTiCrN	CrN	DLC
Base pressure [Pa]	5×10^{-3}	5×10^{-3}	5×10^{-3}	1×10^{-3}
Working pressure [Pa]	4.5	2.5	4	2
Substrate bias voltage [V]	-40	-50	-50	-500
Target current [A]	Cr – 70 AlSi – 120	TiAl – 120 Cr - 140	Cr - 125	-
Process temperature [ºC]	460	430	420	220

Methods of investigations

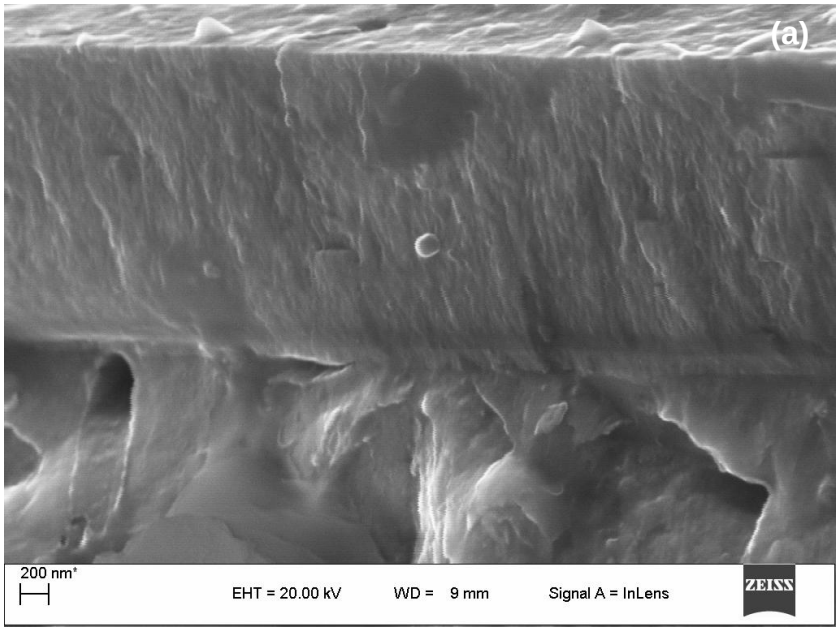
- **Metallographic examinations:**
  - ✓ scanning electron microscope;
  - ✓ high resolution transmission electron microscope;
- **Investigations of the chemical and phase composition:**
  - ✓ spectrometer GDOS;
  - ✓ X-ray diffraction (GAXRD);
  - ✓ Raman spectroscopy;
  - ✓ X-ray Photoelectron Spectroscopy (XPS);

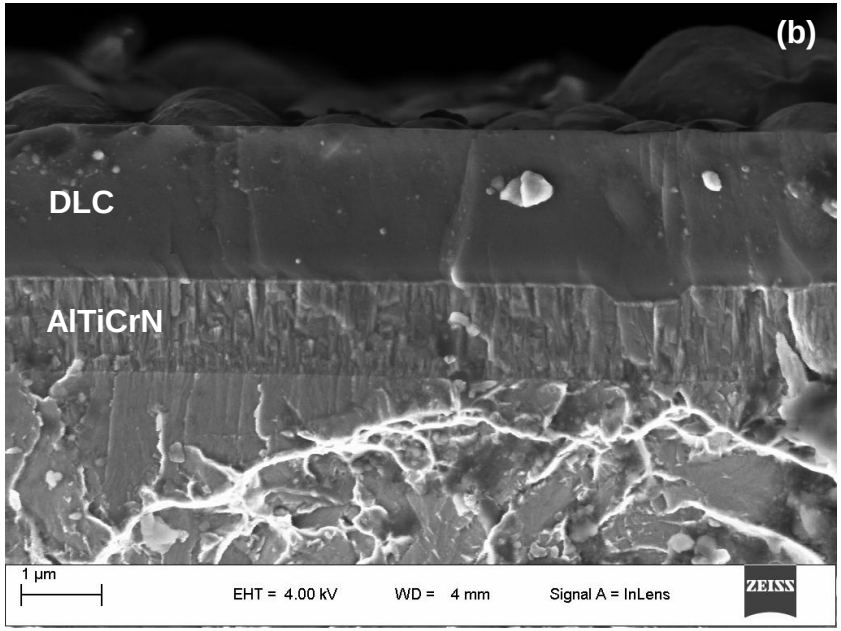
Methods of investigations

- **Mechanical properties:**
  - ✓ microhardness;
  - ✓ adhesion test (scratch test)
  - ✓ tribological test (ball-on-disc);

- **Working properties:**
  - ✓ corrosion test;
  - ✓ extrusion test;

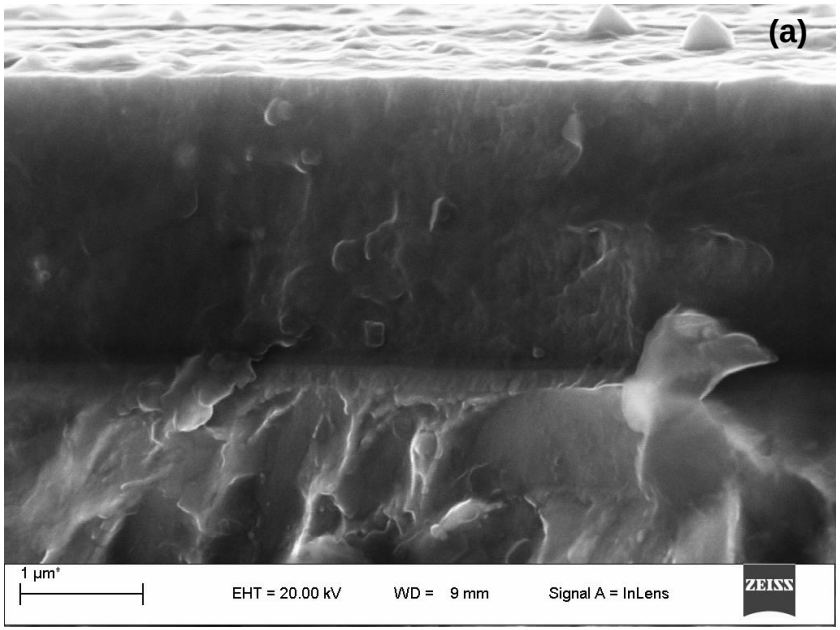
Structures of the coatings

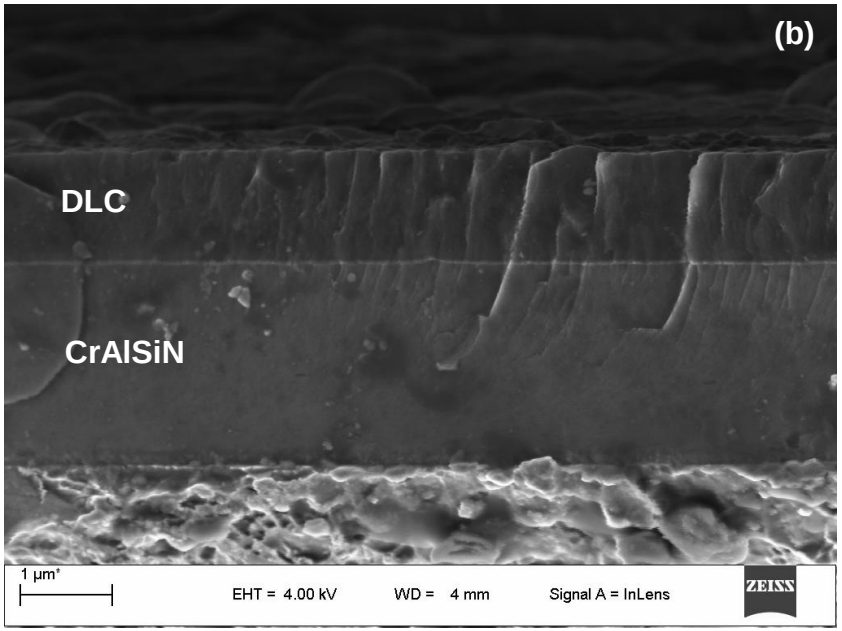




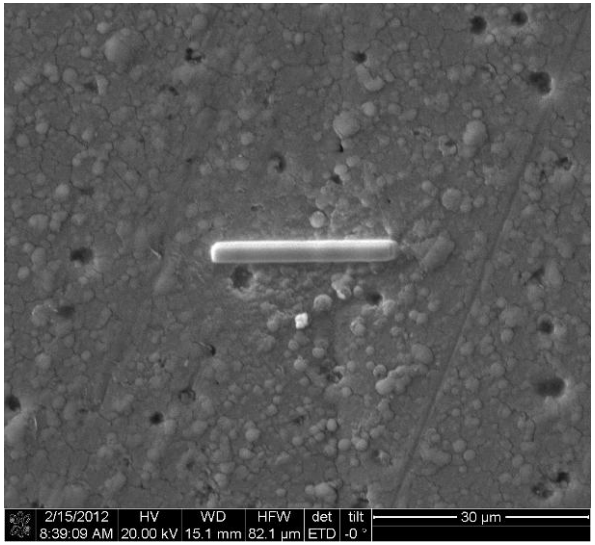
Cross sectional morphology of the: a) AlTiCrN, b) AlTiCrN+DLC coating deposited onto the X40CrMoV5-1 steel substrate

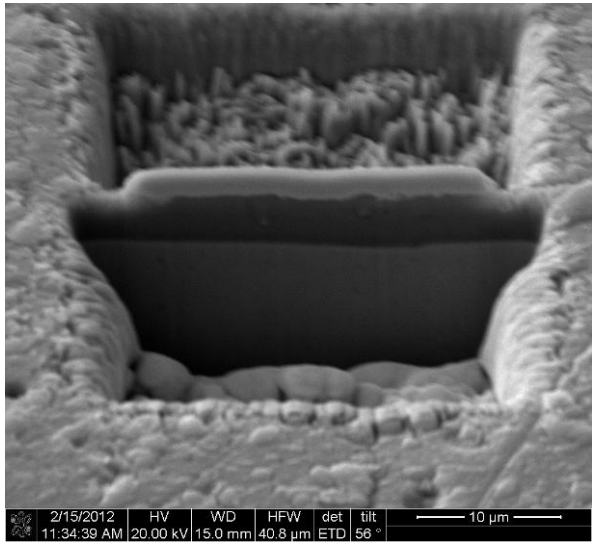
Structures of the coatings

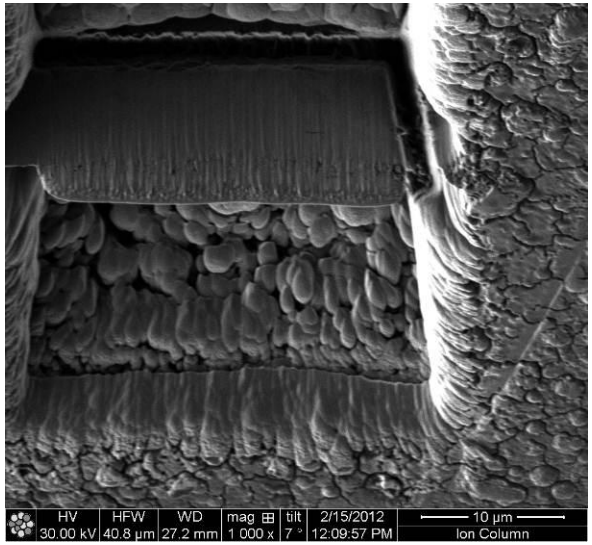


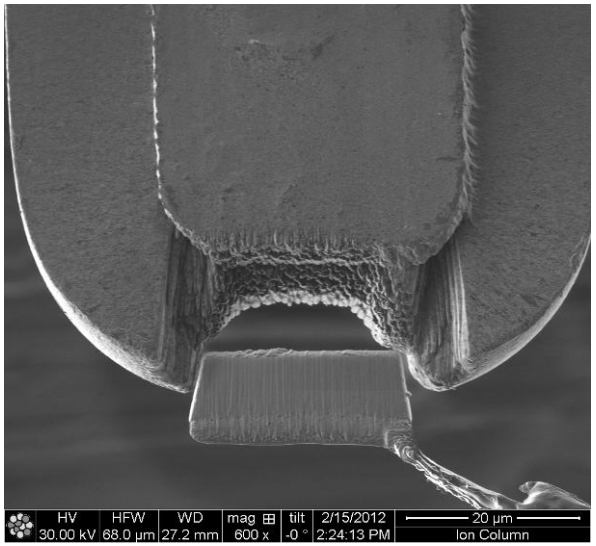


Cross sectional morphology of the: a) CrAlSiN, b) CrAlSiN+DLC coating deposited onto the X40CrMoV5-1 steel substrate

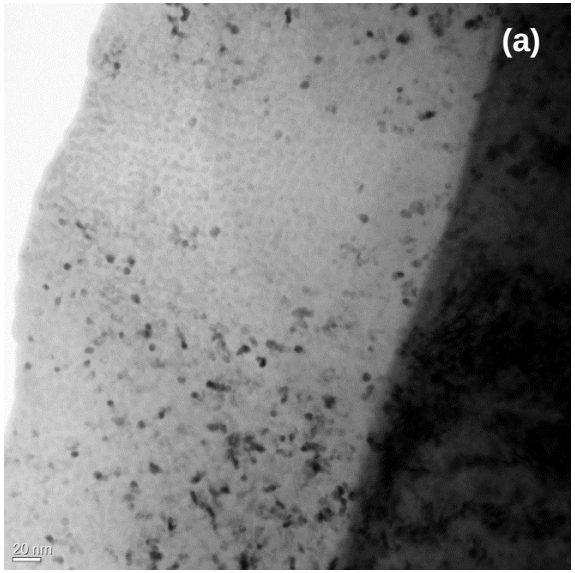


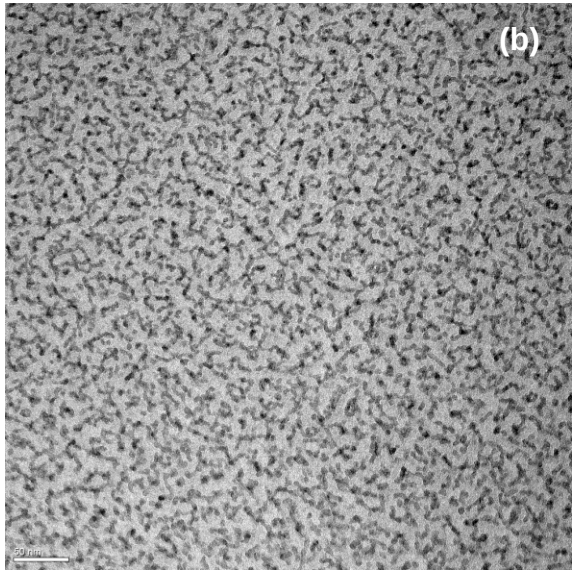


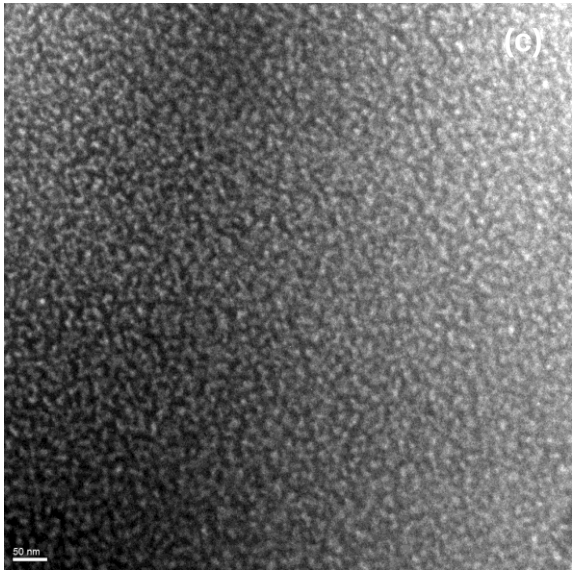


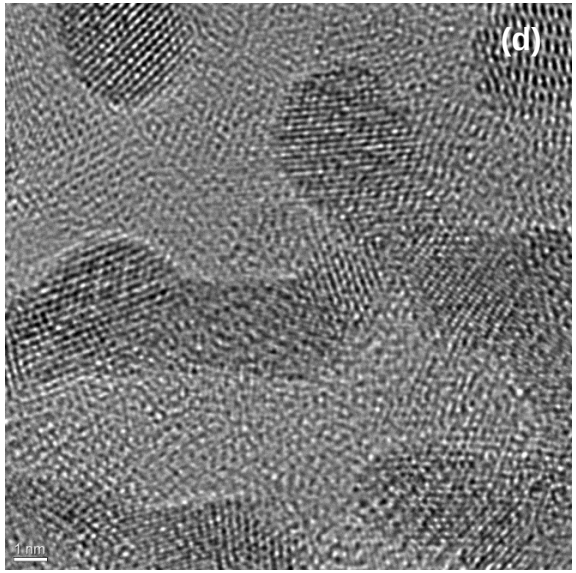


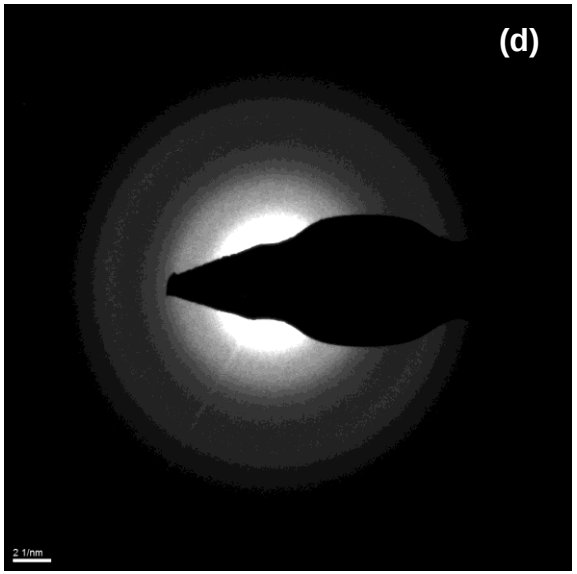
TEM lamella preparation procedure



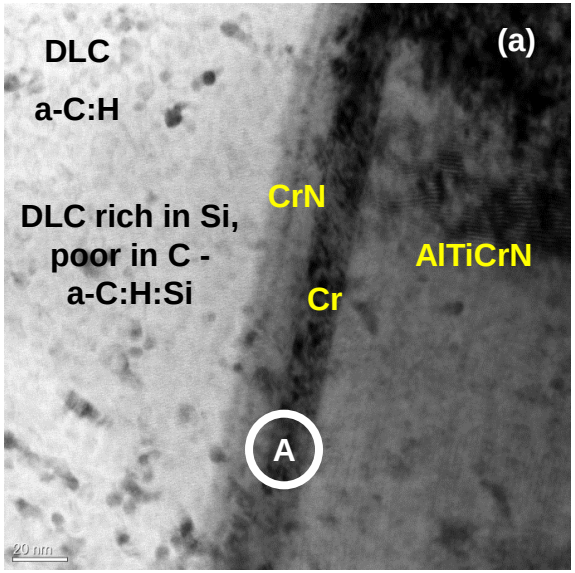


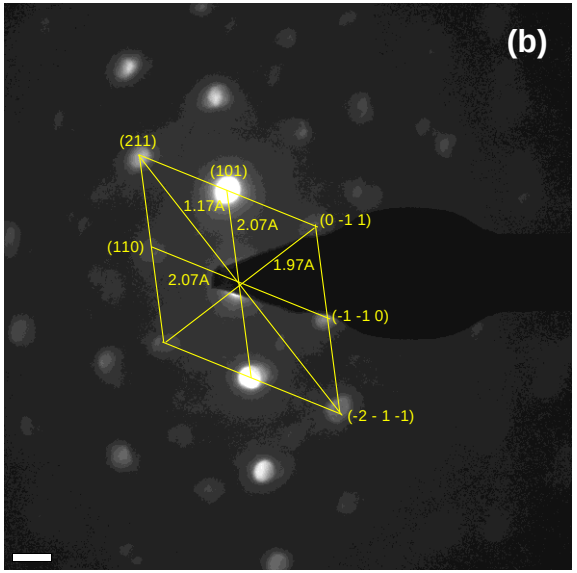


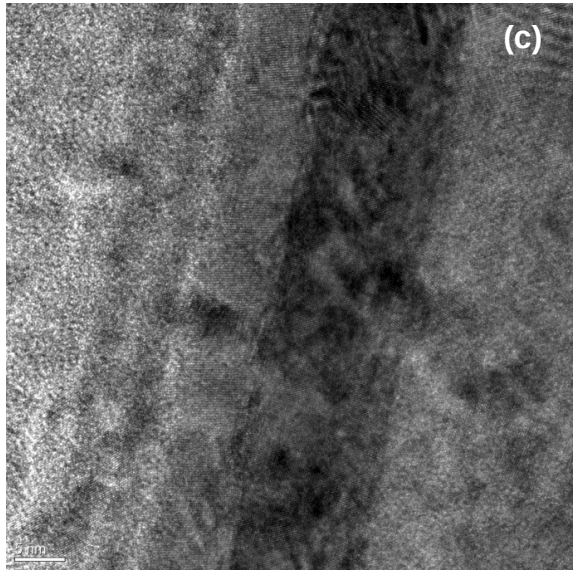


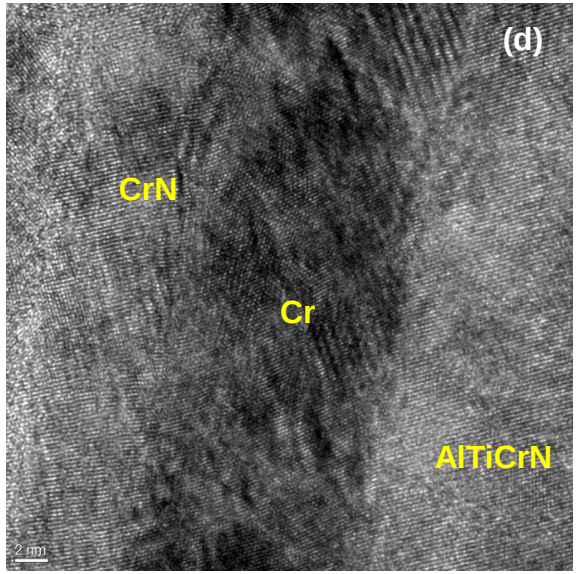


DLC coating: a) TEM cross–section FIB lamella, b) bright field, c) dark field, d) HRTEM, e) SAD

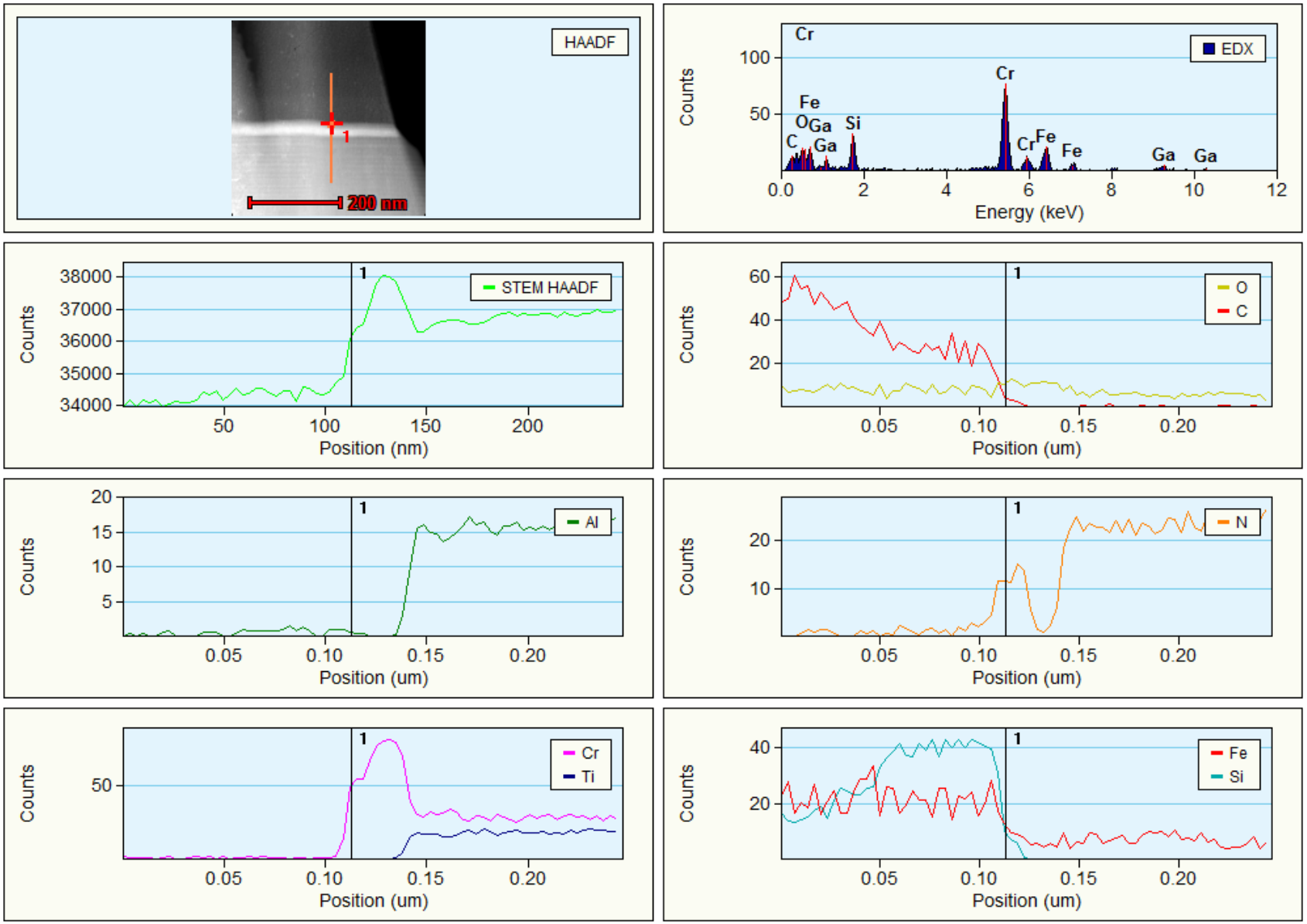


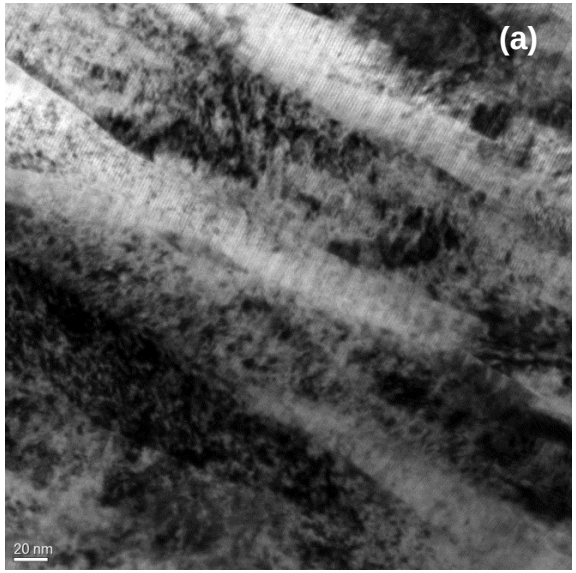


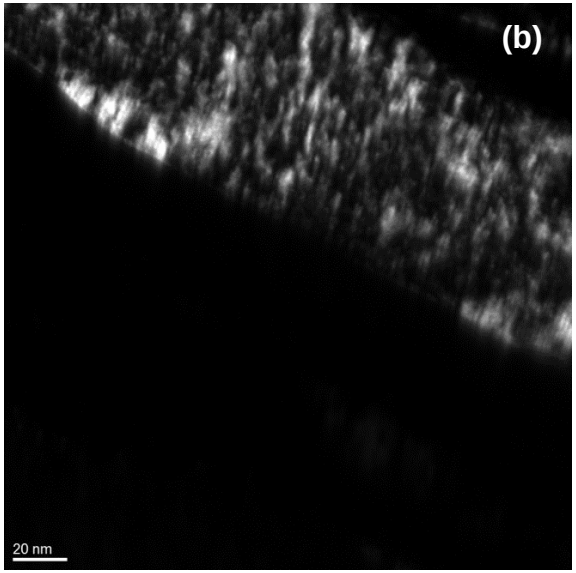


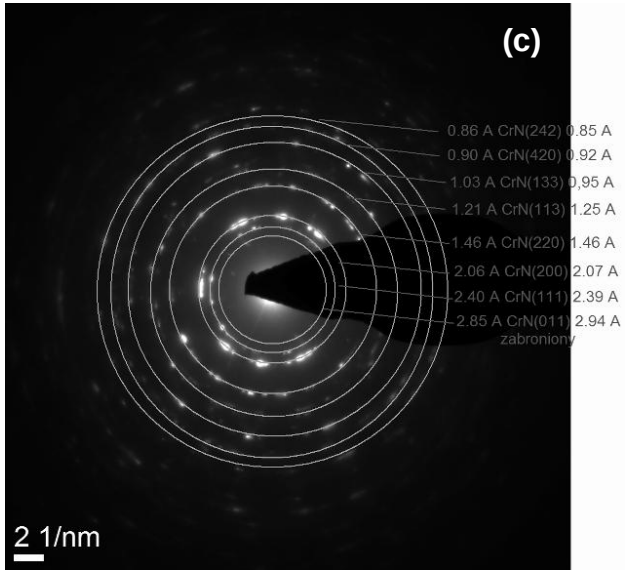


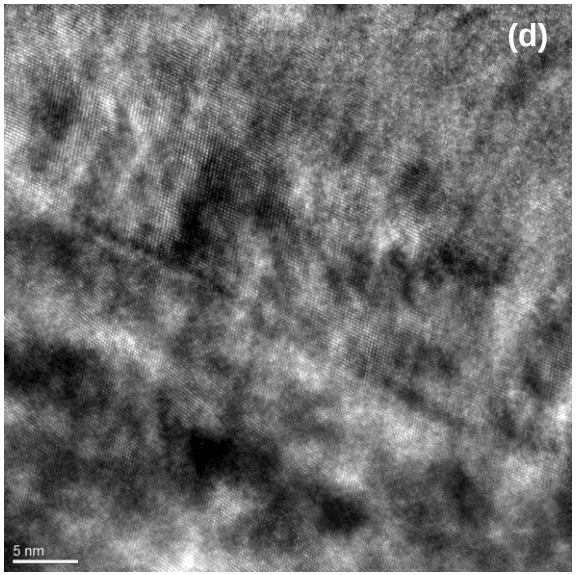
Microstructure of the transition zone between topcoat DLC and AlTiCrN layer: a) TEM micrograph, b) SAD pattern from the area A, c, d) HRTEM micrograph

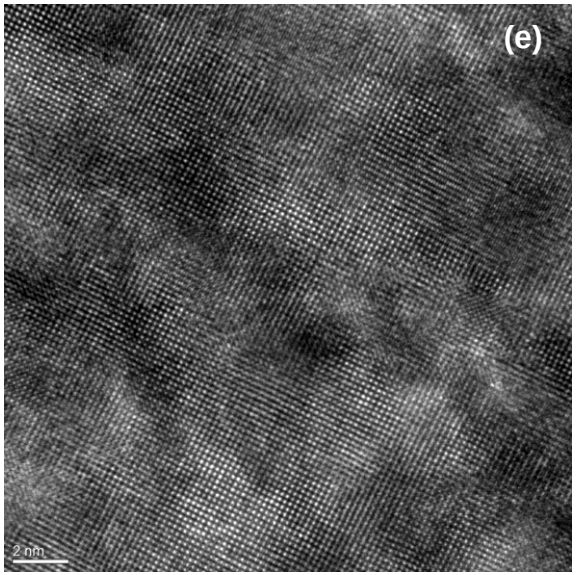

HAADF
1
200 nm
EDX
Counts
Energy (keV)
Cr
Fe
O
Ga
C
Si
STEM HAADF
Position (nm)
Position (um)
O
C
Al
N
Ti



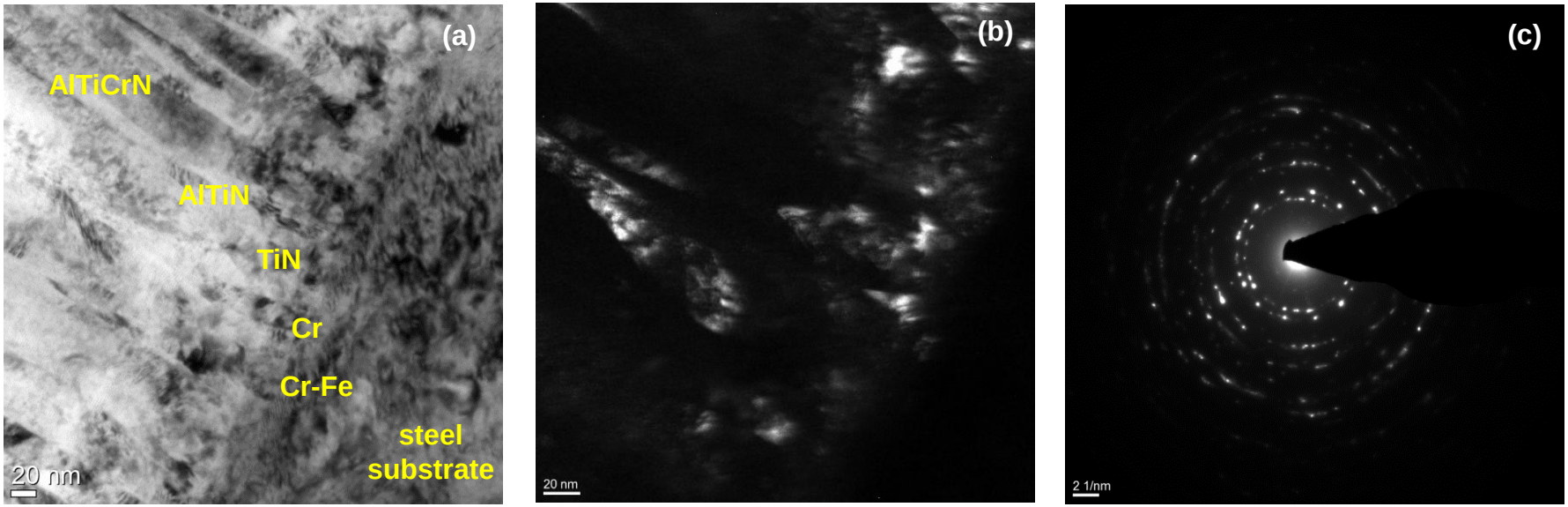




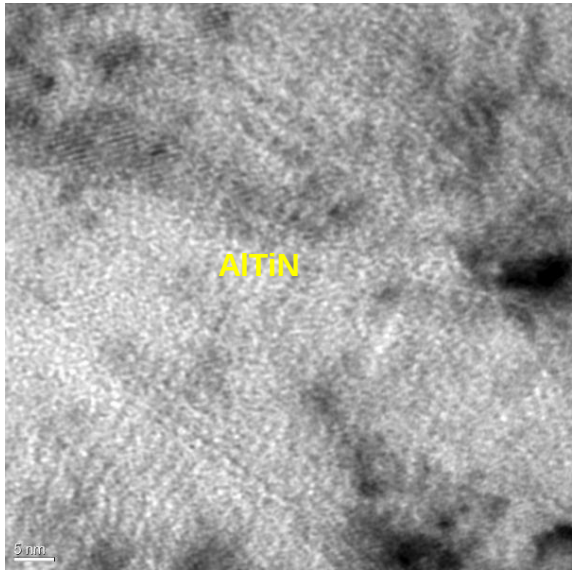


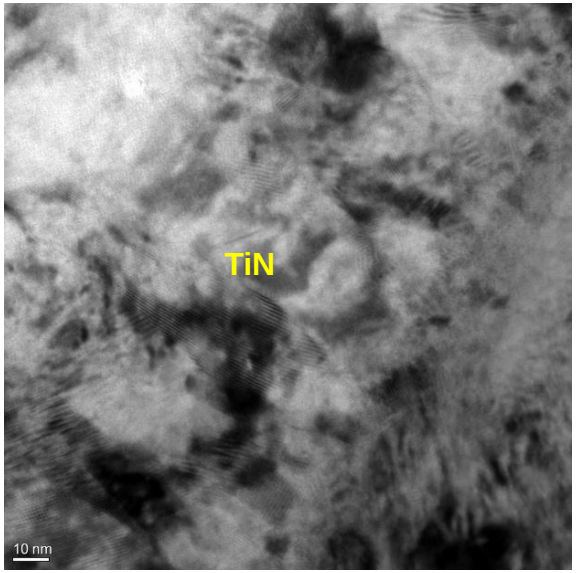


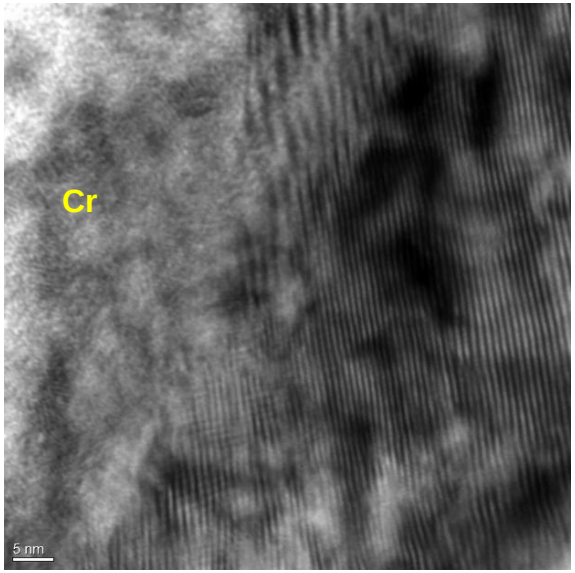
AlTiCrN coating: a) bright field, b) dark field, c) SAD, d, e) HRTEM

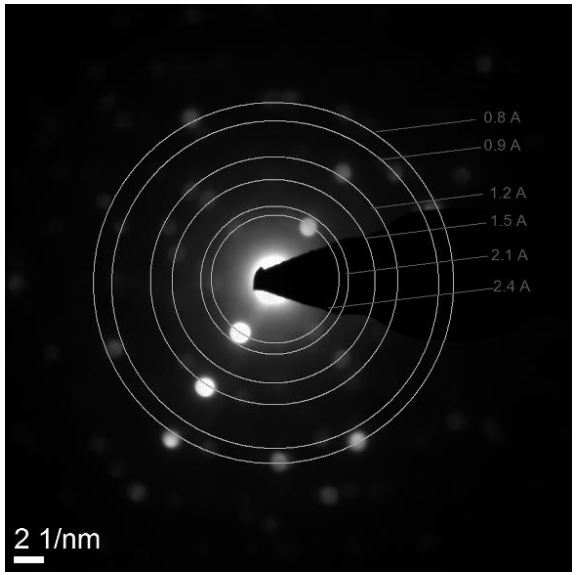


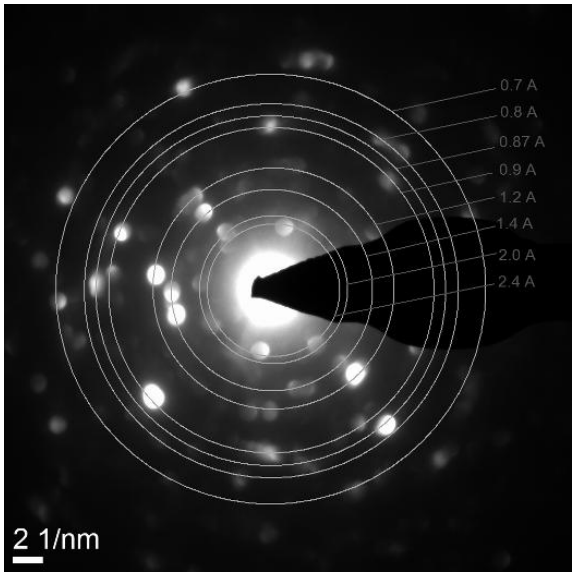
TEM micrograph of the transition zone between AlTiCrN layer and steel substrate: a) bright field, b) dark field, c) SAD pattern from the area as in figure a

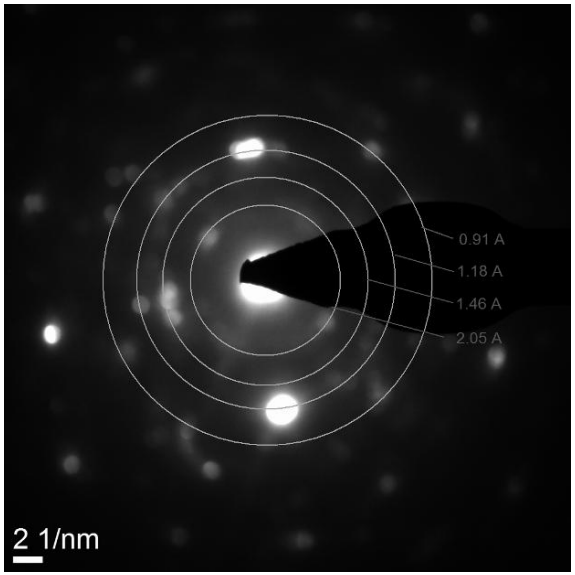

AlTiN
5 nm

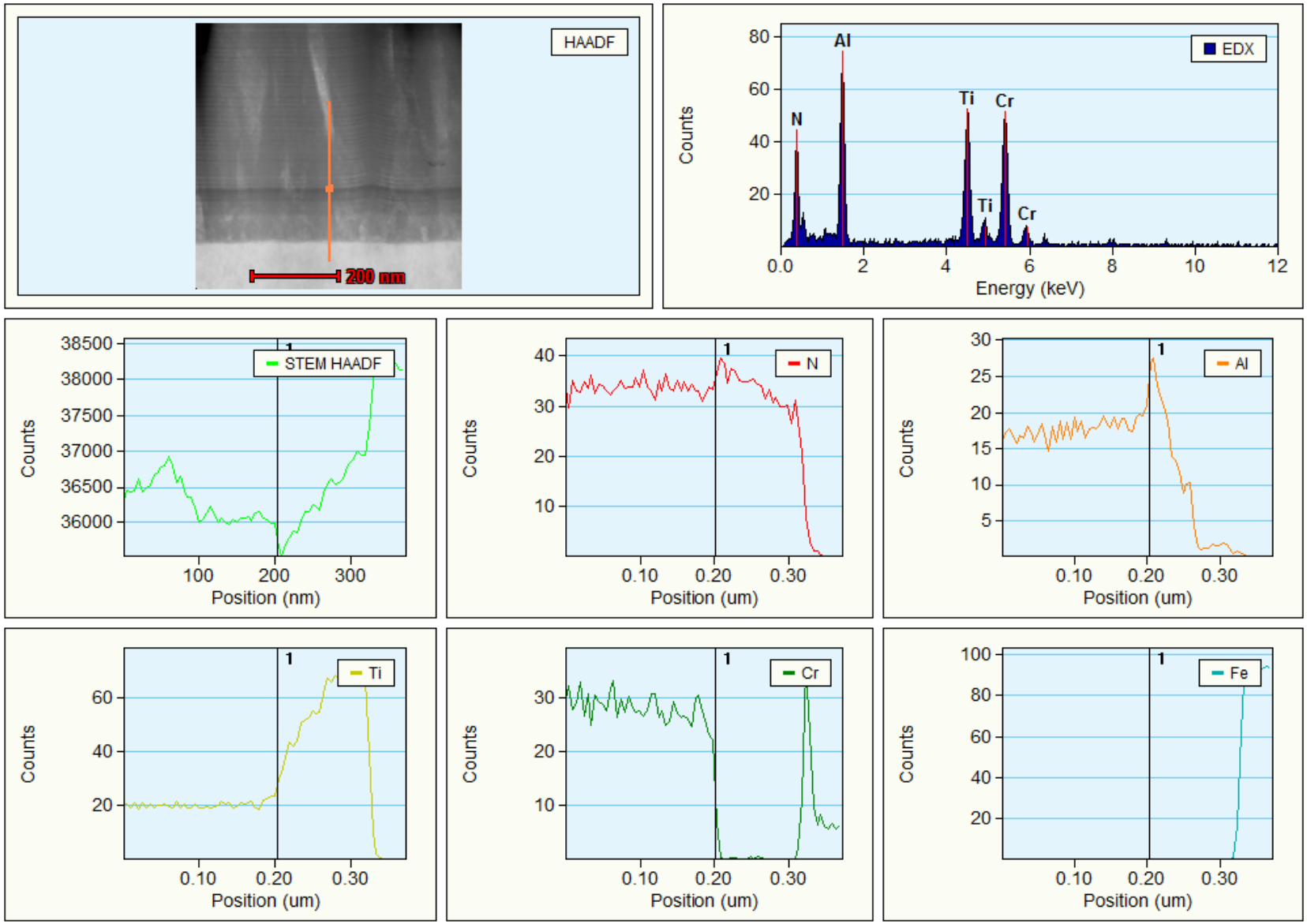

TiN
10 nm


Cr
5 nm

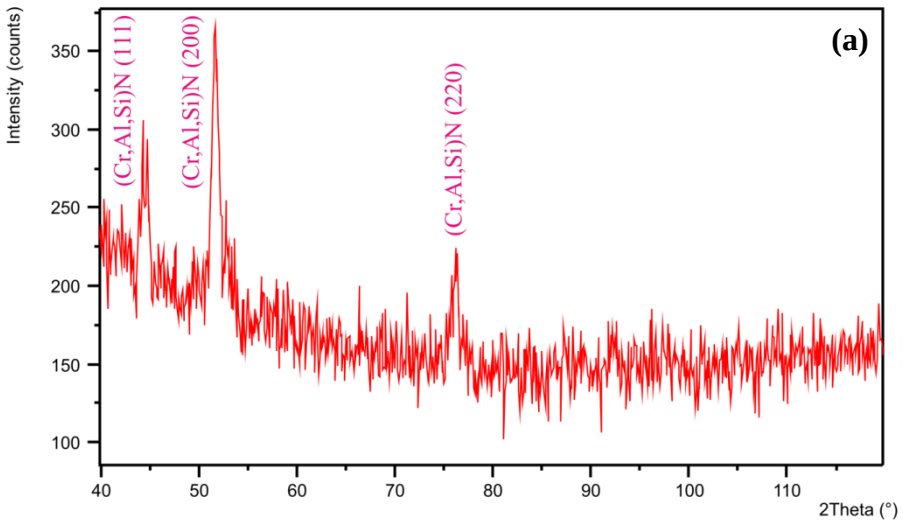

0.8 A
0.9 A
1.2 A
1.5 A
2.1 A
2.4 A
2 1/nm

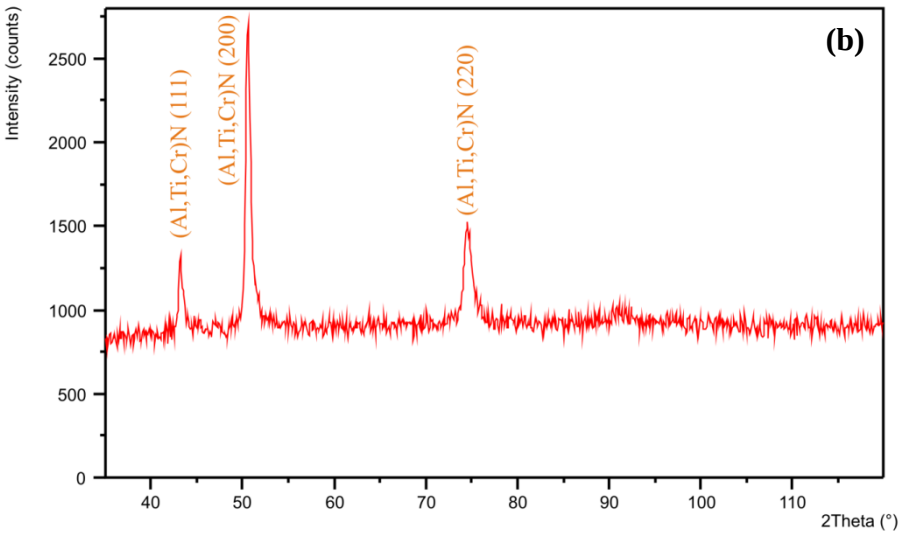

0.7 A
0.8 A
0.87 A
0.9 A
1.2 A
1.4 A
2.0 A
2.4 A
2 1/nm


0.91 A
1.18 A
1.46 A
2.05 A
2 1/nm

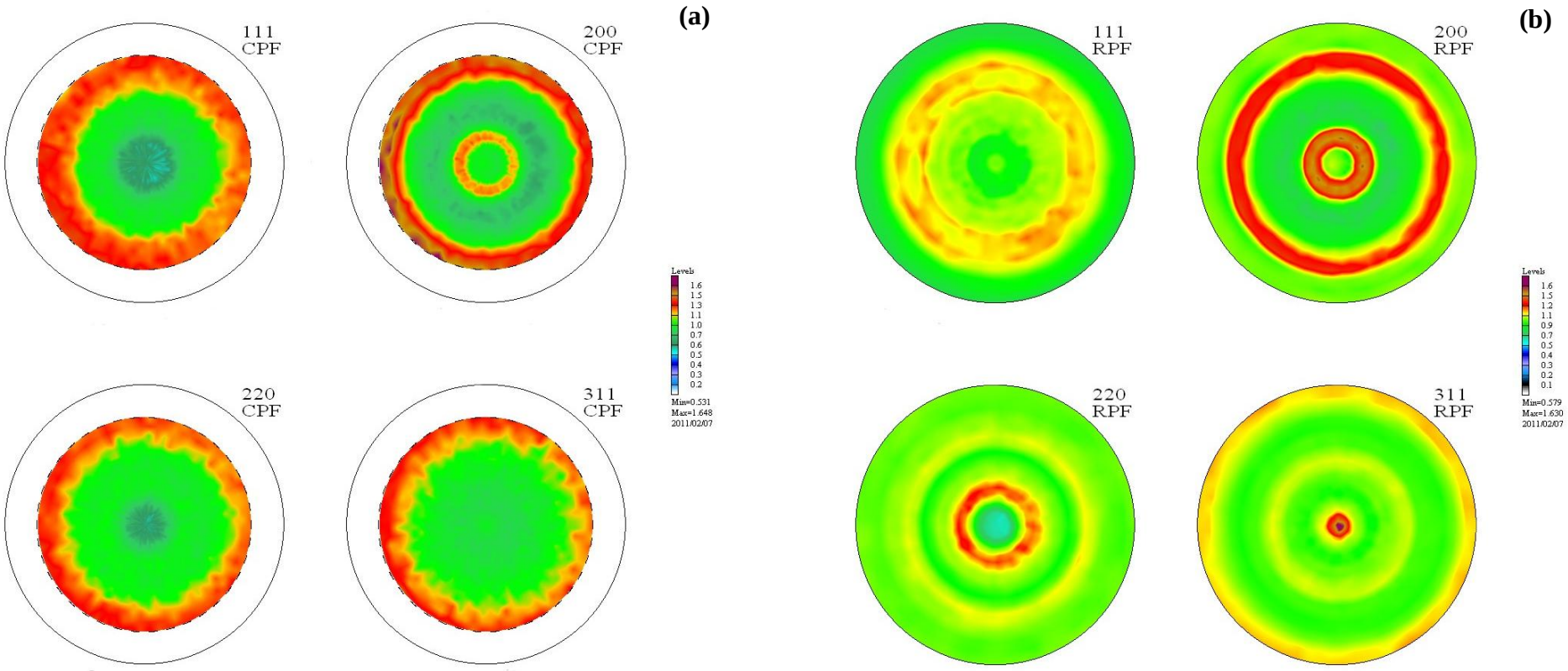

HAADF
200 nm
EDX
Counts
Energy (keV)
N
Al
Ti
Cr
STEM HAADF
Position (nm)
Position (um)
Fe

X-ray diffraction



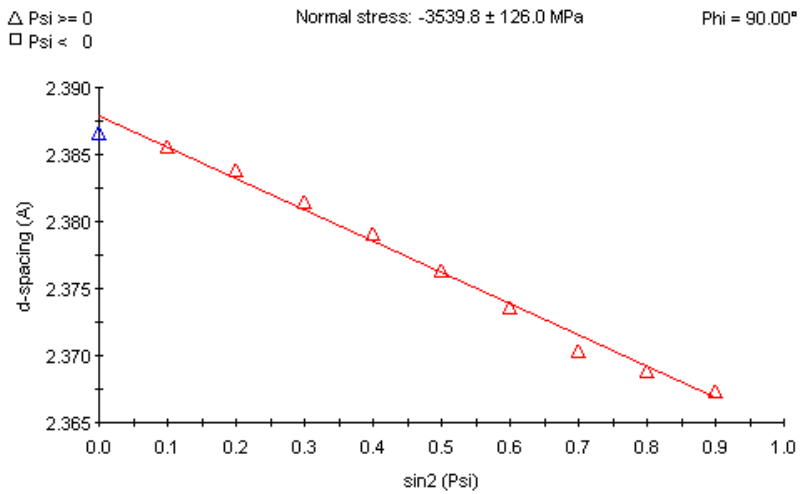


GAXRD spectra of the: a) CrAlSiN, b) AlTiCrN coating at glancing incidence angle α=2°

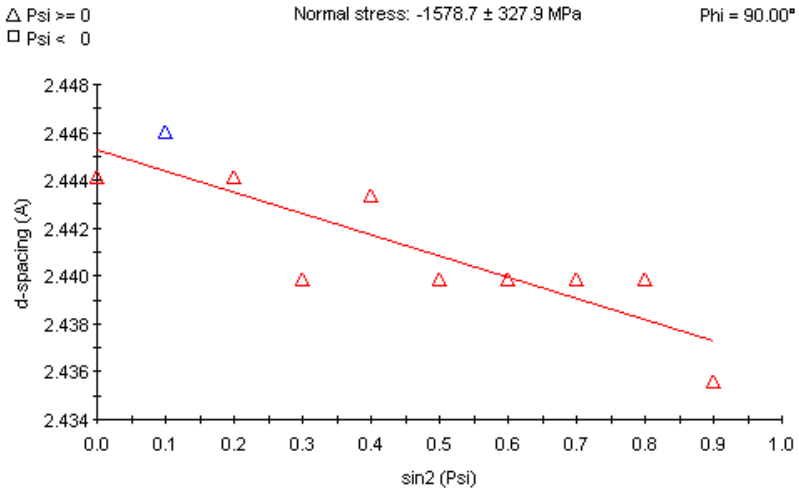


Pole figures (111), (200), (220) and (311) of CrAlSiN coating deposited onto X40CrMoV5-1 steel substrate: a) experimental, b) calculation based on FRO

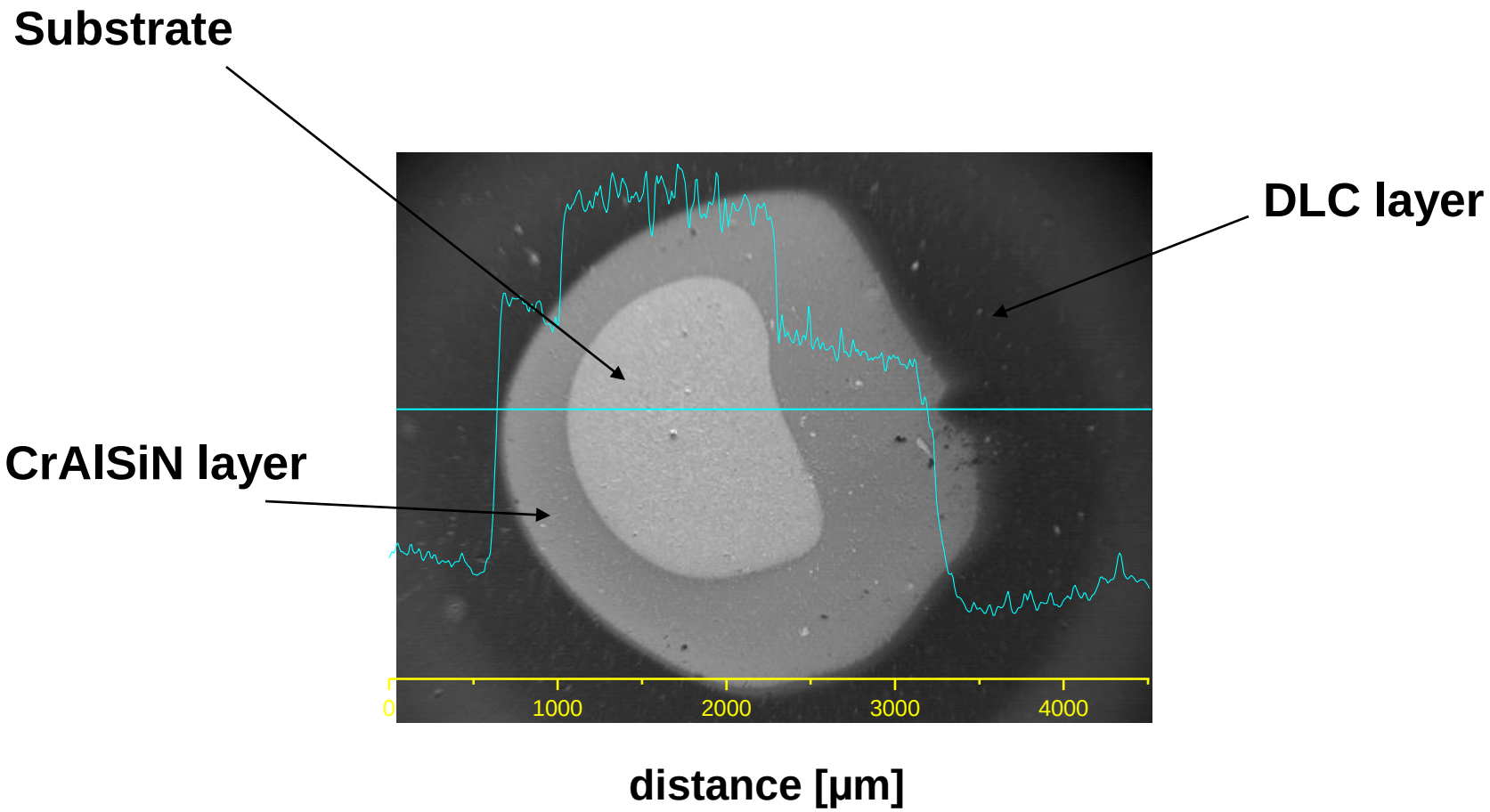
(a)



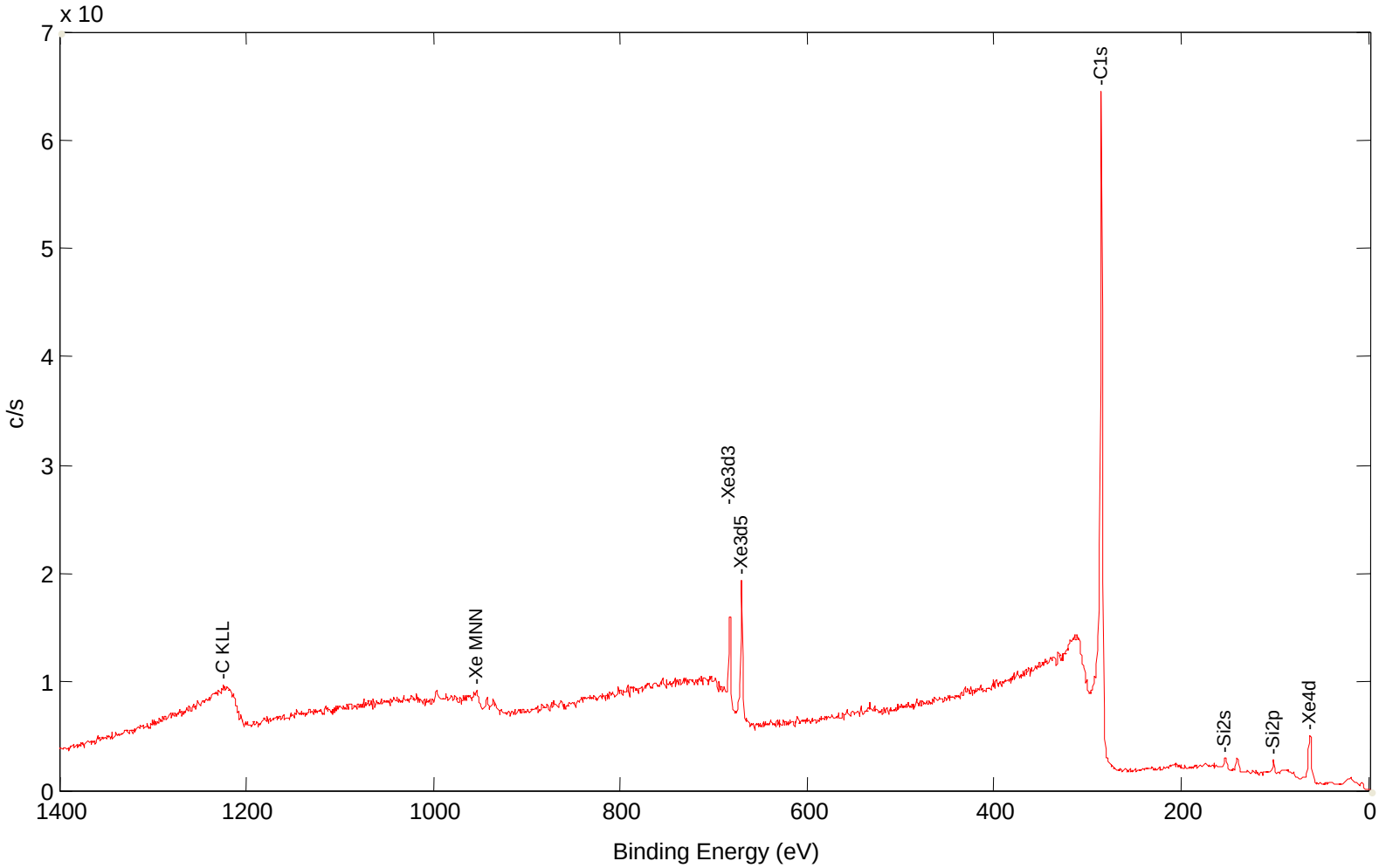
(b)



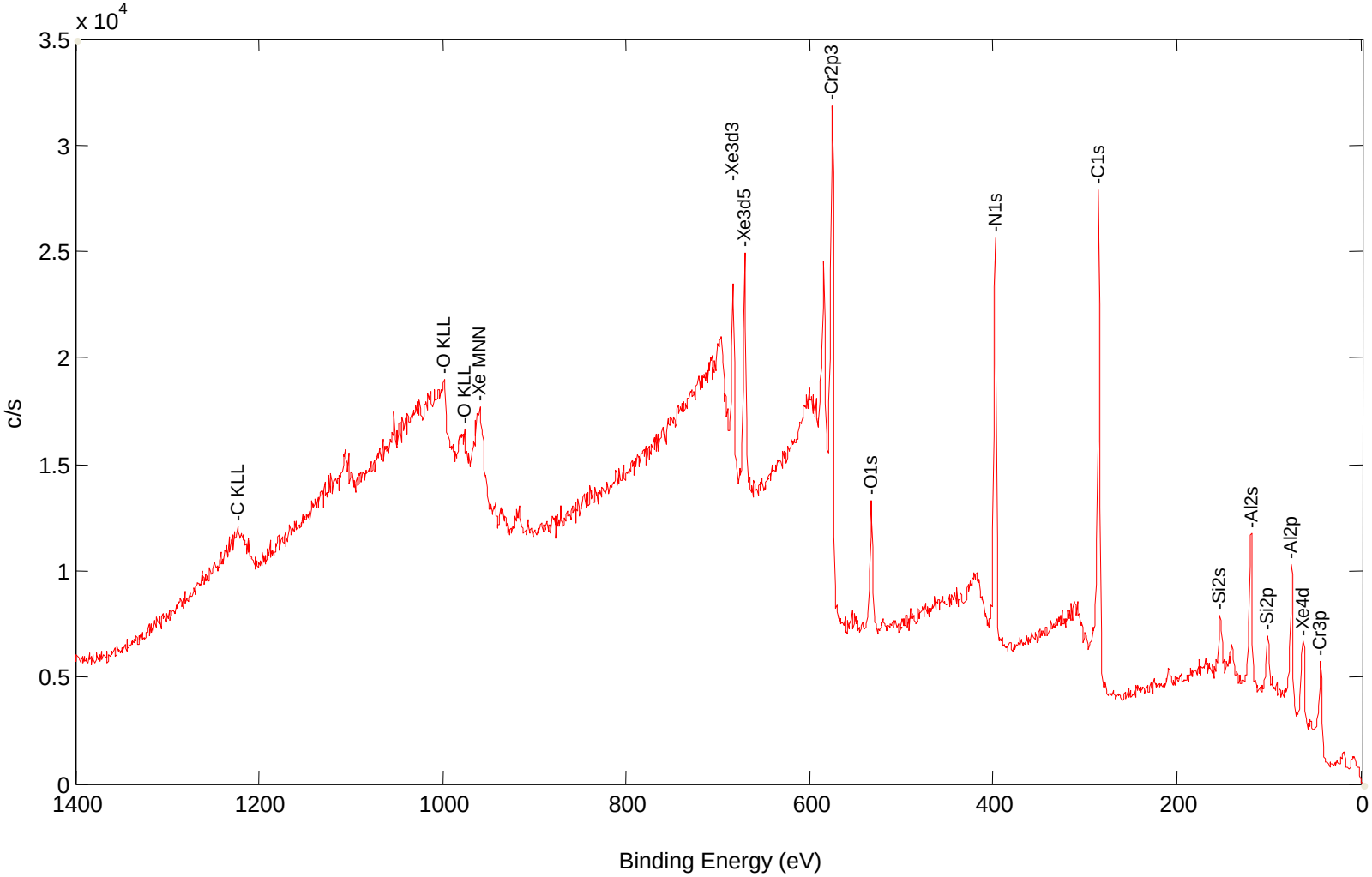
Changes of interplanar distance value d in the a) CrAlSiN, b) CrN layer as a function of sin2ψ (stresses` measurement made by sin2ψ method for different φ values of samples setting towards goniometer axis)



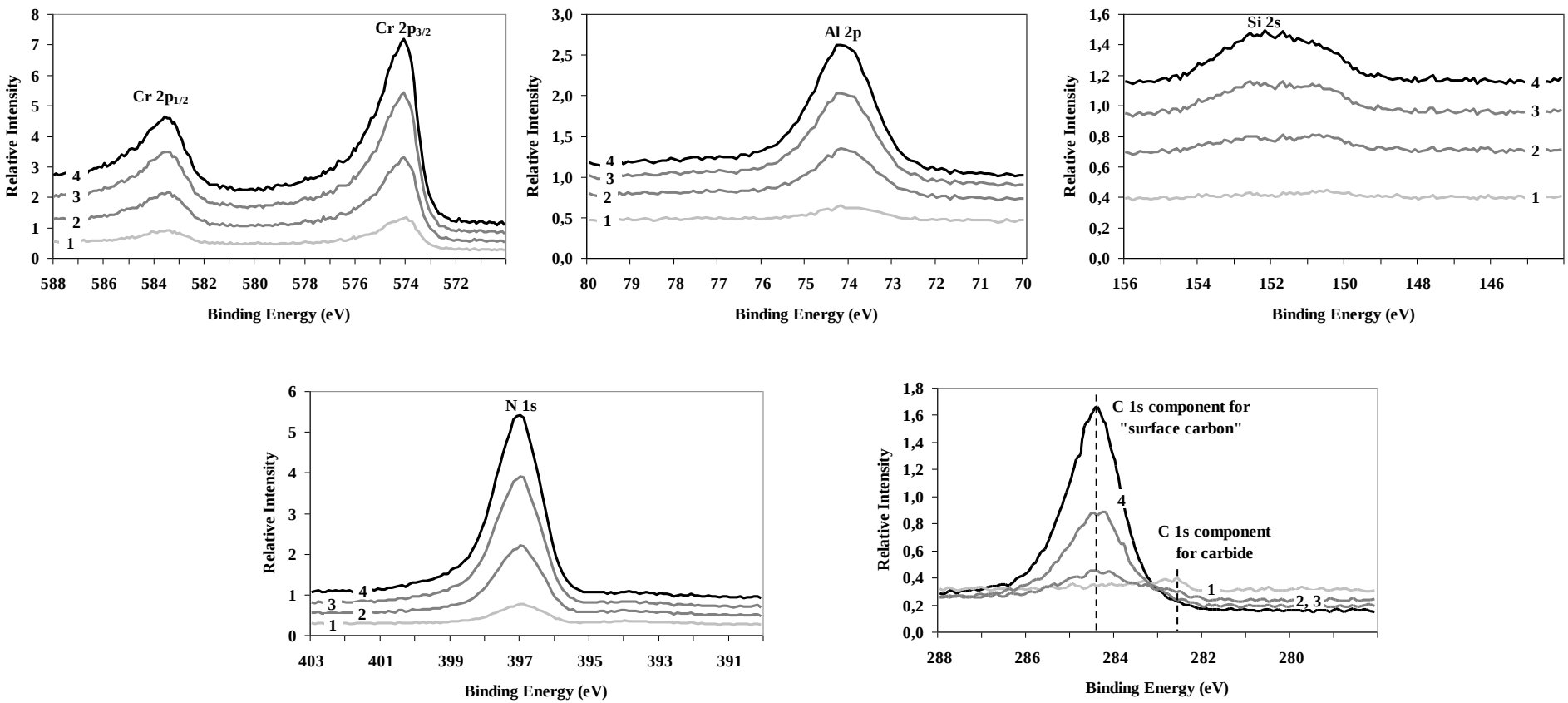
SEM picture of area with sputter crater of CrAlSiN+DLC coating



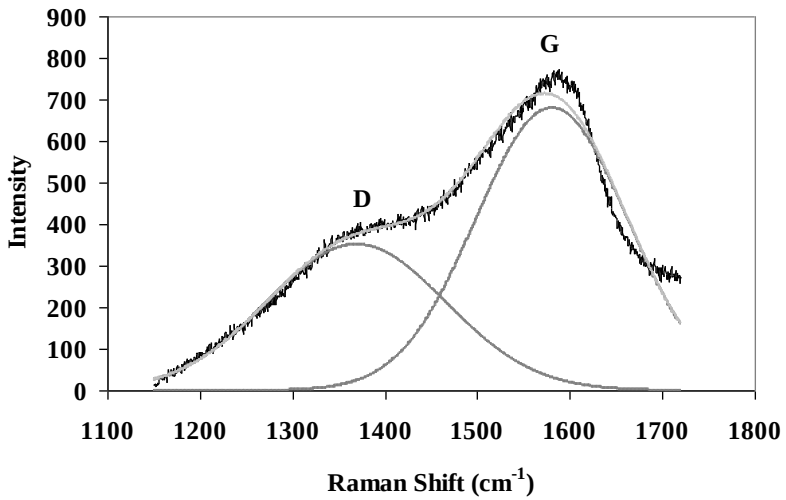
XPS spectra obtained from the DLC layer

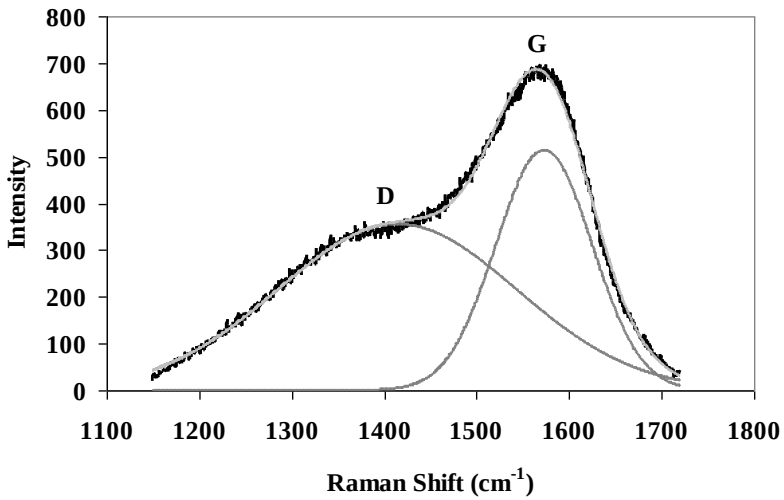


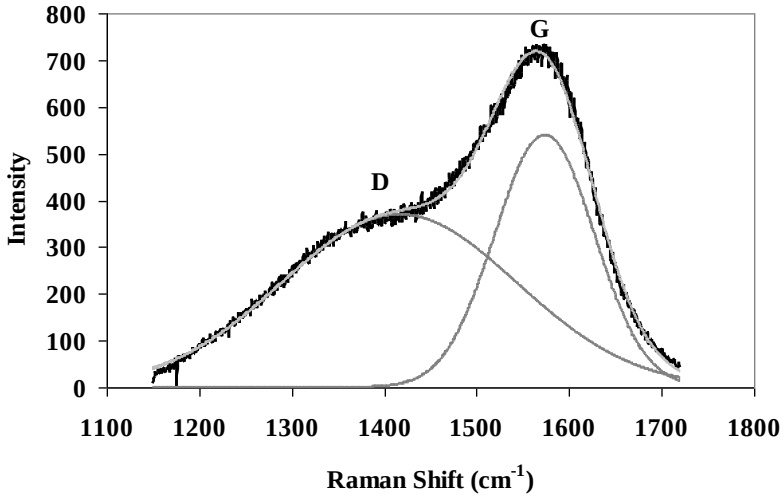
XPS spectra obtained from the CrAlSiN layer



XPS spectra of CrAlSiN+DLC coating at varying surface region: (1) substrate, (2, 3) CrAlSiN film, (4) DLC film; (a) Cr 2p, (b) Al 2p, (c) Si 2s, (d) N 1s, (e) C 1s







Raman spectra of DLC layers deposited on the: (a) CrAlSiN, (b) AlTiCrN, (c) CrN coating

The characteristics of the tested coatings

Coating	Thickness [µm]	Microhardness [GPa]	Crystallite size [nm]	Critical load L_{C1} [N]	Critical load L_{C2} [N]
AlTiCrN +DLC	1.2 1.8	34 20	15	24	54
CrAlSiN +DLC	2.1 1.2	40 20	10	18	49
CrN +DLC	0.5 1.0	24 20	7	16	46

Adhesion

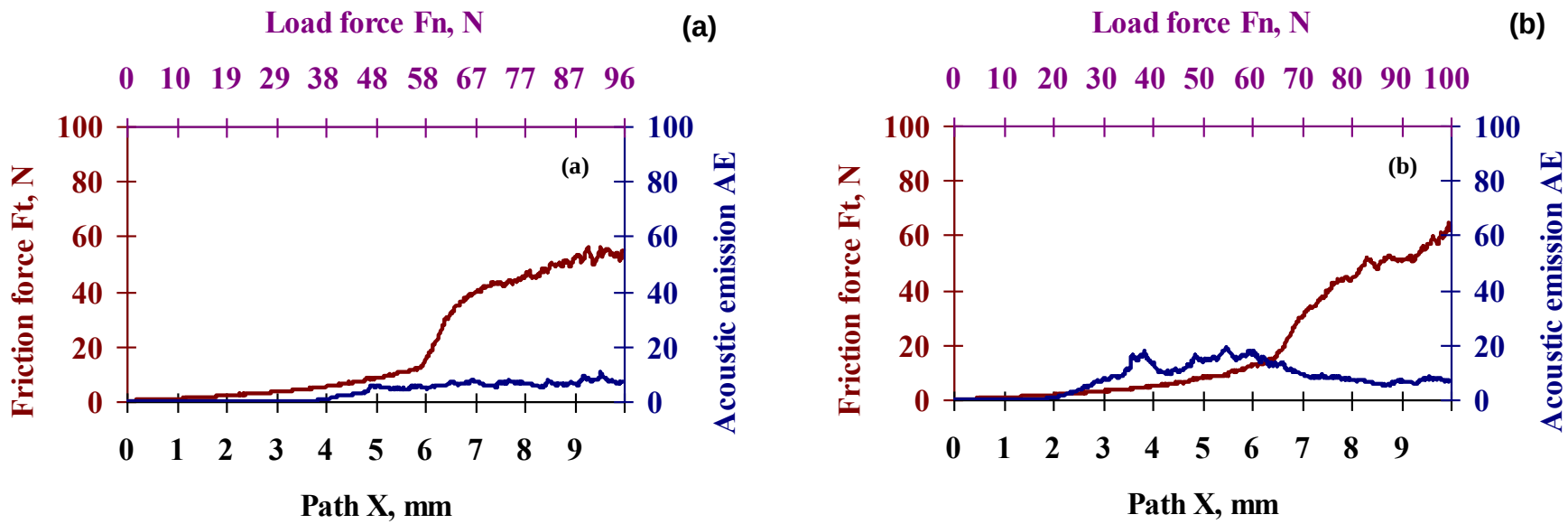


Diagram of the dependence of the acoustic emission (AE) and friction force Ft on the load for the X40CrMoV5-1 steel with the: a) AlTiCrN+DLC, b) CrN+DLC coating

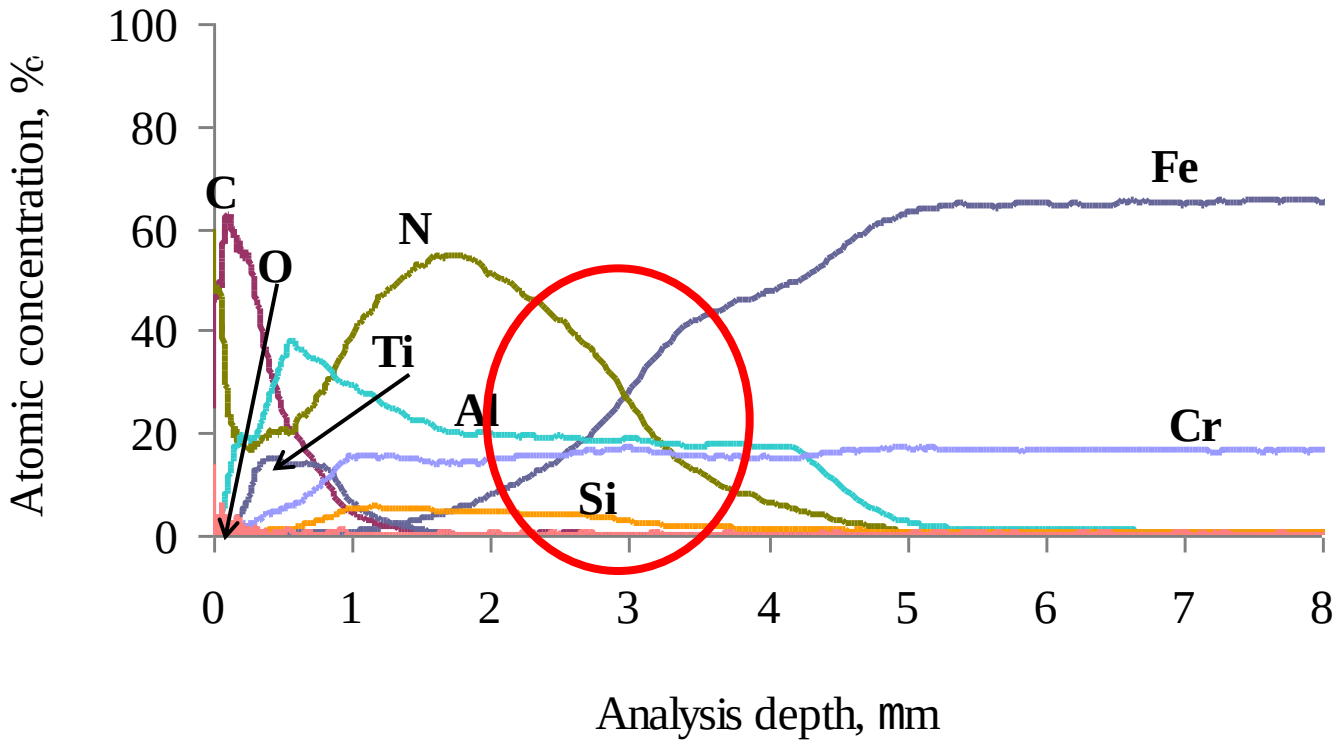
Adhesion

Scratch failure pictures of the AlTiCrN+DLC coating on X40CrMoV5-1 steel substrate at: (a) L_{C1}, (b) L_{C2}

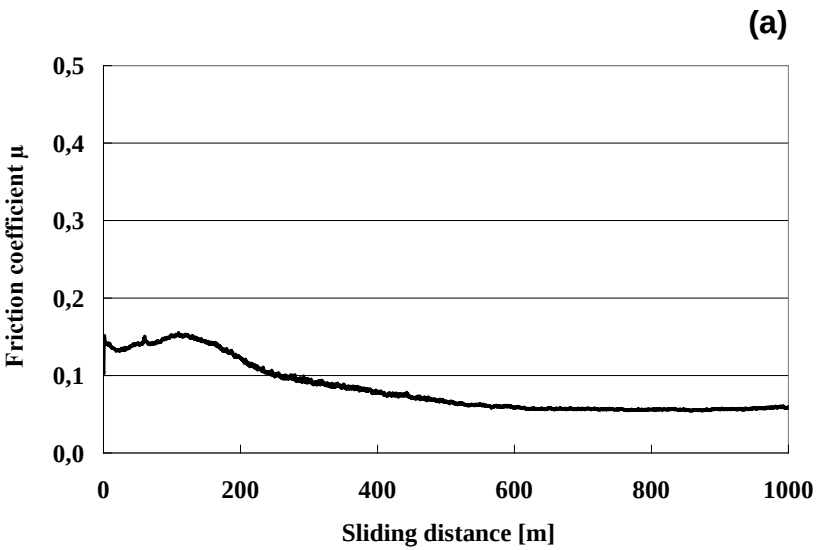
The characteristics of the tested coatings

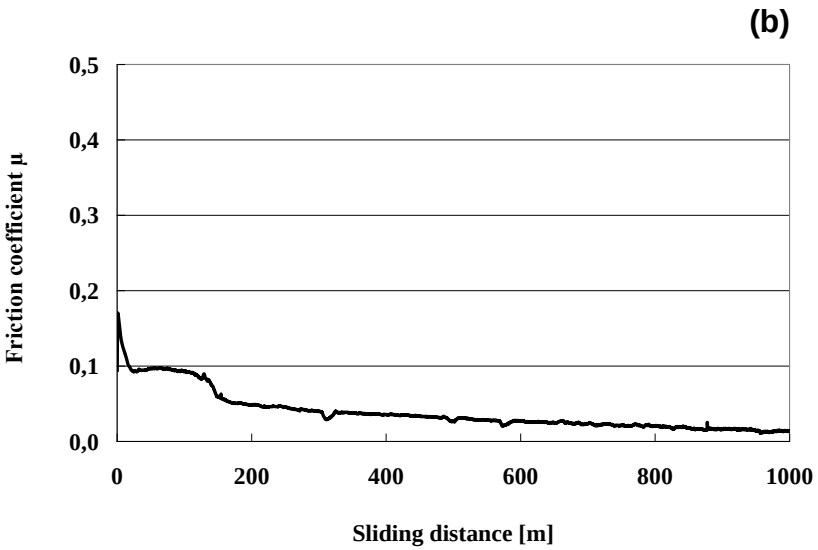
Coating	Thickness [µm]	Microhardness [GPa]	Crystallite size [nm]	Critical load L_{C1} [N]	Critical load L_{C2} [N]
AlTiCrN +DLC	1.2 1.8	34 20	15	24	54
CrAlSiN +DLC	2.1 1.2	40 20	10	18	49
CrN +DLC	0.5 1.0	24 20	7	16	46

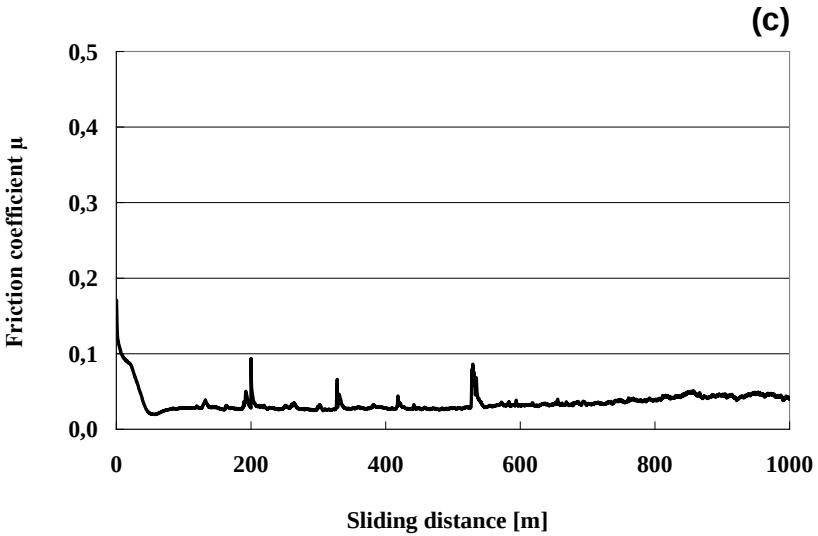
Chemical composition



Changes of constituent concentration of the CrAlSiN+DLC coatings and the substrate materials

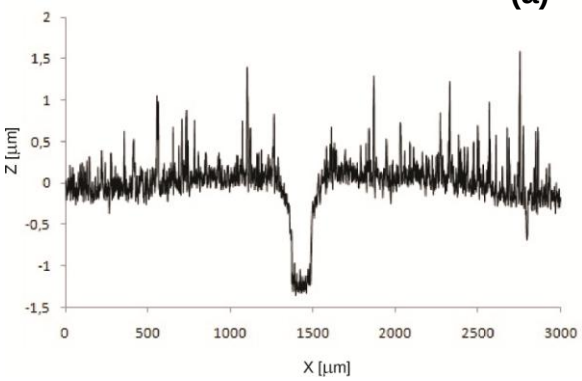




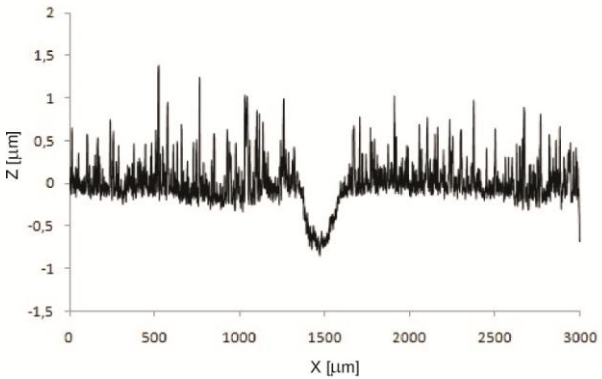


Dependence of friction coefficient on sliding distance during the wear test for: (a) CrAlSiN+DLC, (b) AlTiCrN+DLC, (c) CrN+DLC coating

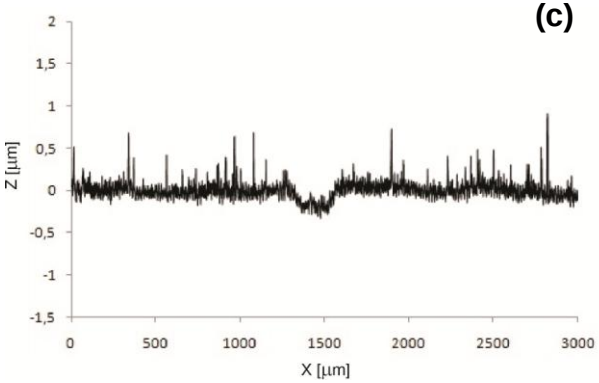
(a)



(b)



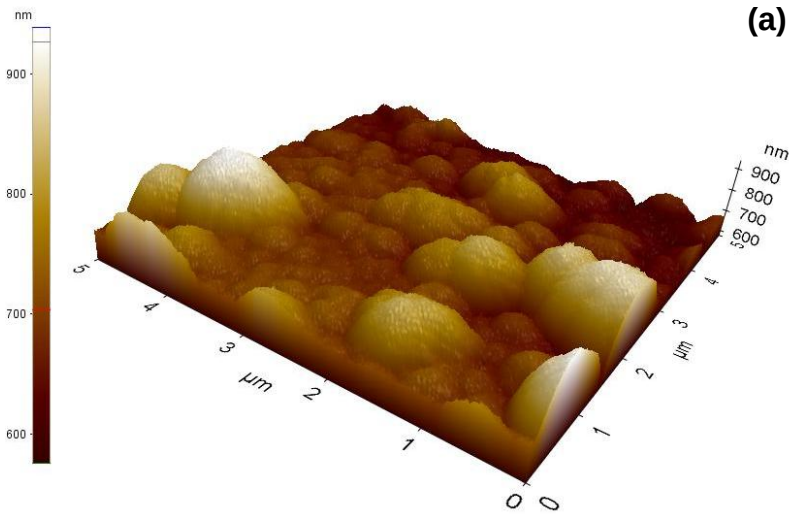
(c)

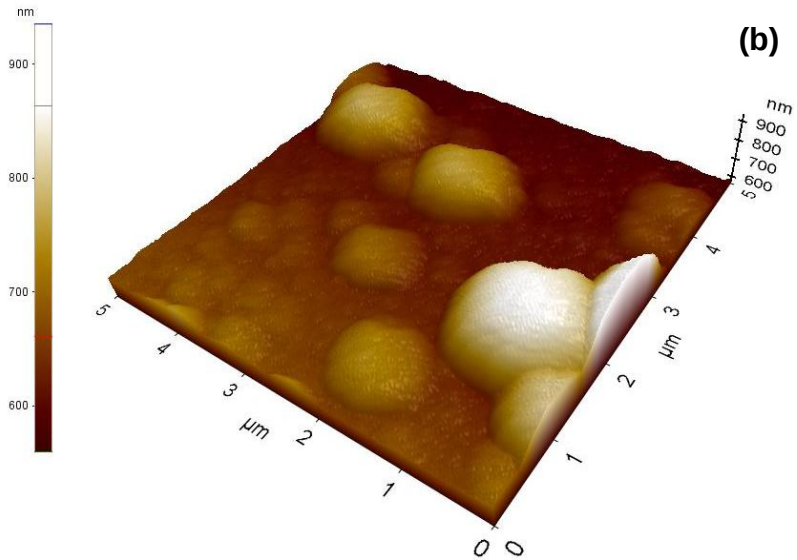


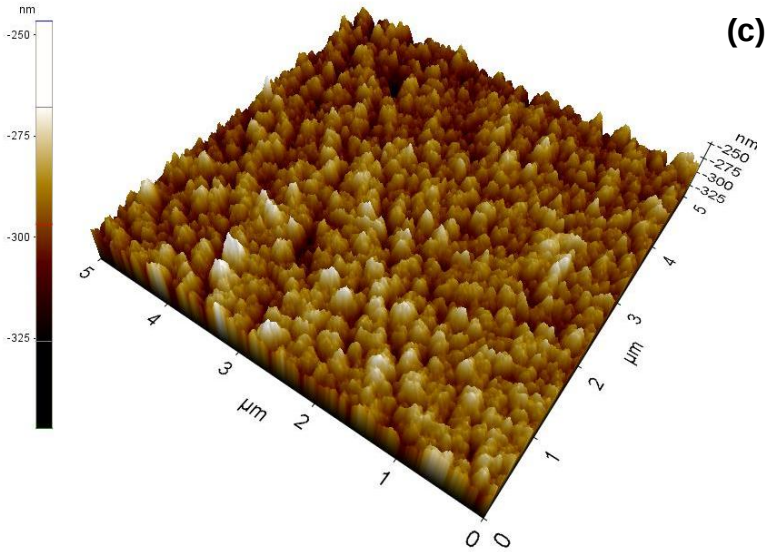
Wear pattern profile after the wear test of: a) CrAlSiN+DLC, b) AlTiCrN+DLC, c) CrN+DLC coating

The characteristics of the tested coatings

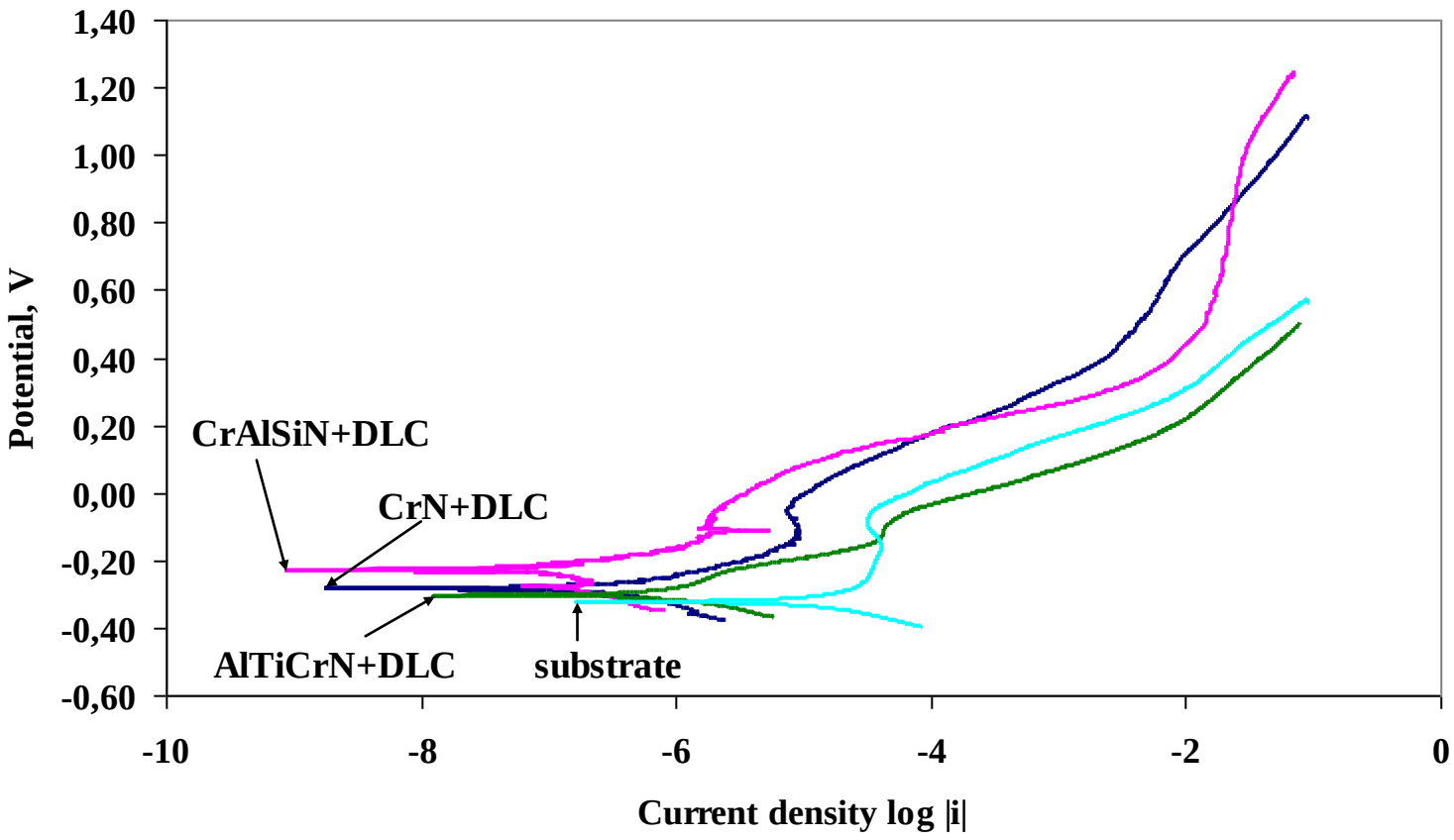
Coating	Friction coefficient μ	Wear rate of coatings k_{vc}, [mm³/Nm]	Wear rate of counterpart k_{vb}, [mm³/Nm]
AlTiCrN +DLC	0.03	3.30×10^{-7}	4.53×10^{-9}
CrAlSiN +DLC	0.06	4.54×10^{-7}	9.71×10^{-9}
CrN +DLC	0.04	1.58×10^{-7}	6.56×10^{-9}



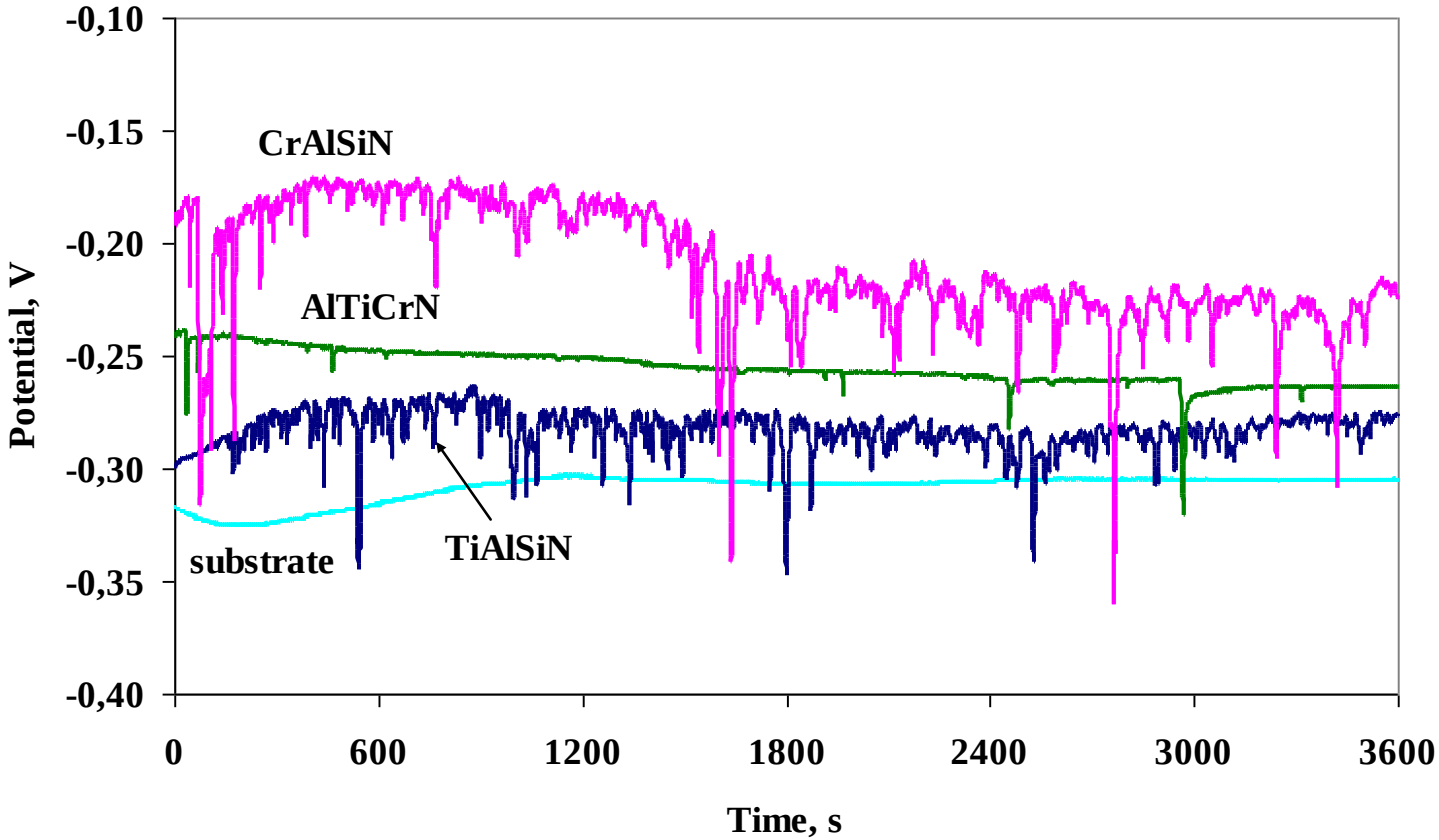




AFM image of: a) CrAlSiN+DLC, b) AlTiCrN+DLC, c) CrN+DLC coating



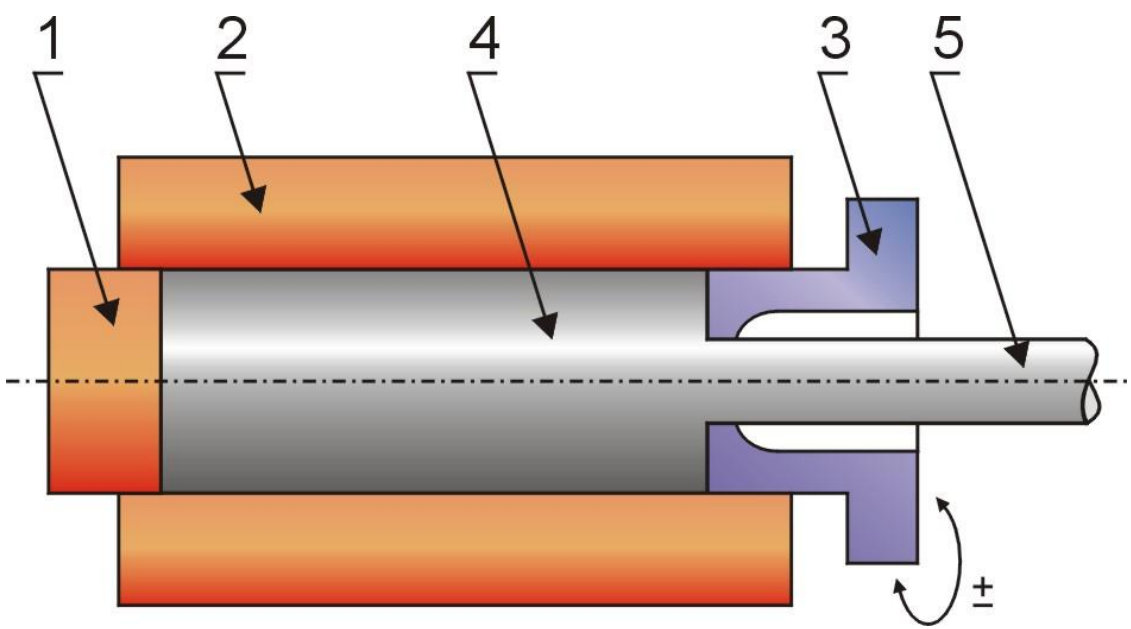
Potentiodynamic polarization curves of the investigated coatings in 1 M HCl solution



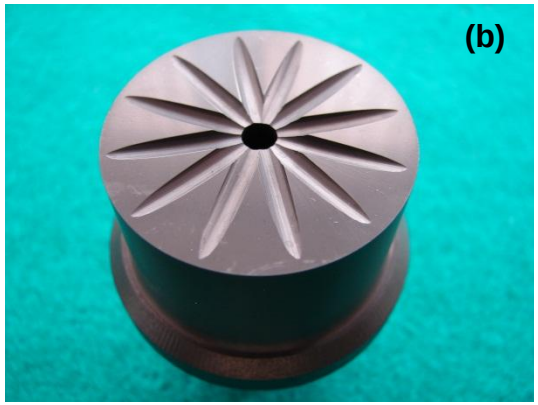
Open circuit potential curves of the investigated coatings in 1 M HCl solution

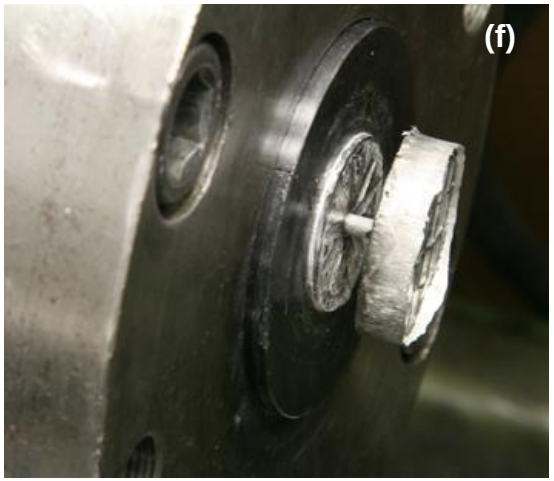
Summary results of the electrochemical corrosion investigation

Coating type	Current density i_{cor}, [$\mu A/cm^2$]	Corrosion potential E_{cor}, [mV]	Corrosion rate, [mm/year]
AlTiCrN	0.77	-0.26	9.0
CrAlSiN	0.15	-0.22	1.7
TiAlSiN	0.48	-0.28	5.6
Substrate	11.65	-0.30	136.2



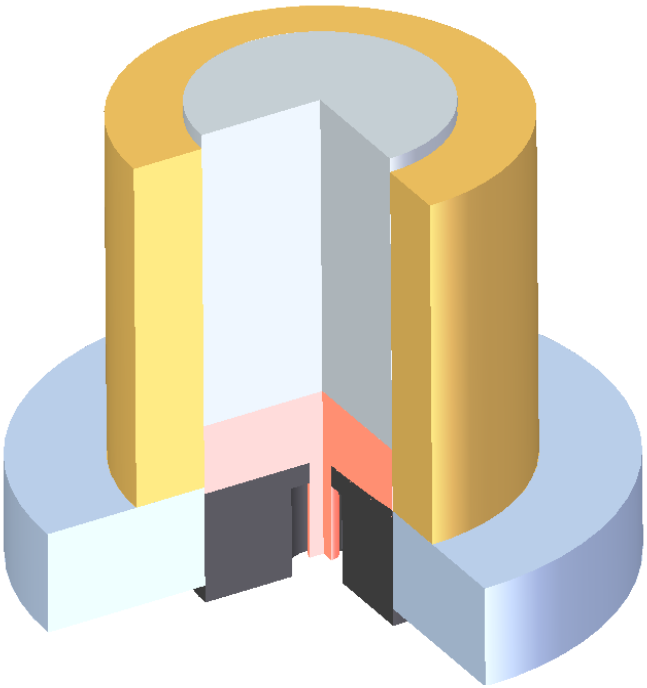
Deformation of metallic materials with the KOBO method in the extrusion process: 1 - punch, 2 - container, 3 - reversaly rotated die, 4 - extruded material, 5 - product



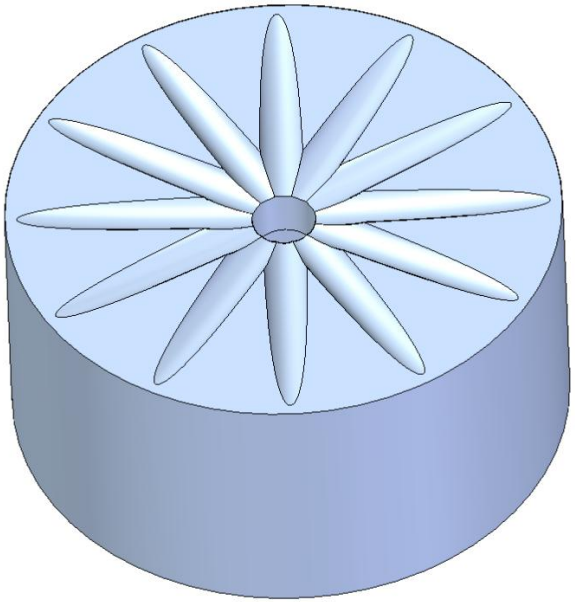


Example of the typical appearance of the die coated by CrN+DLC coating before (a, b, c) and after (d, e, f) extrusion test

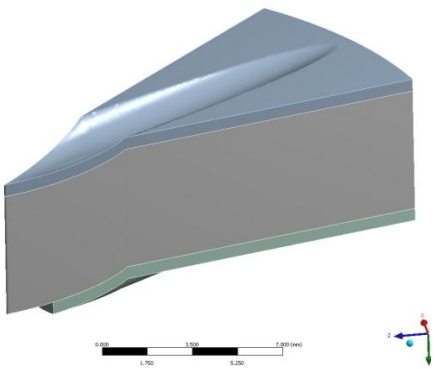
(a)

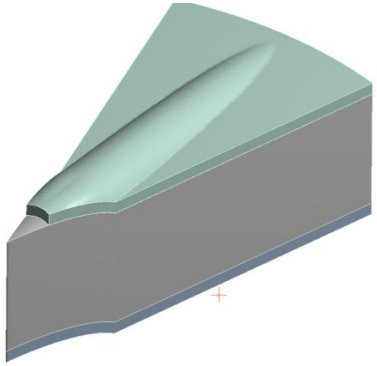


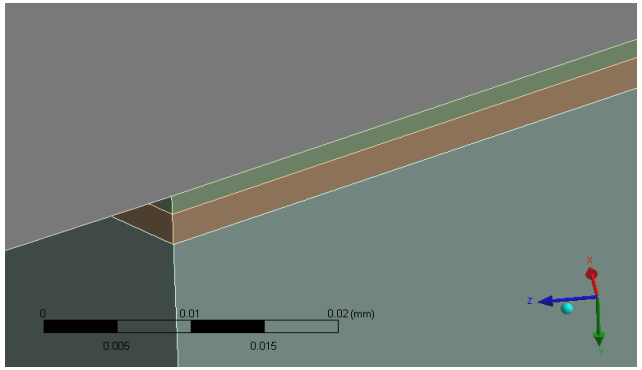
(b)

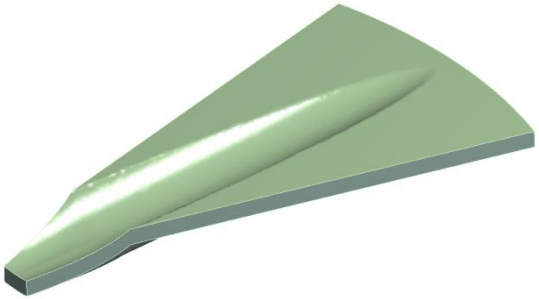


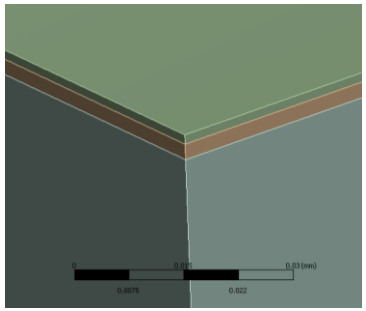
CAD model of the: (a) extrusion process, (b) die



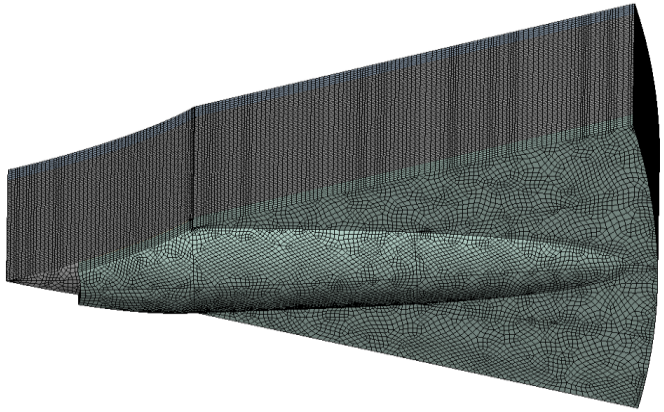


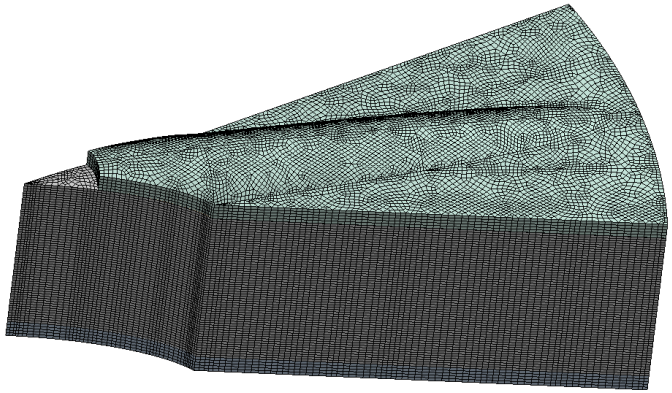


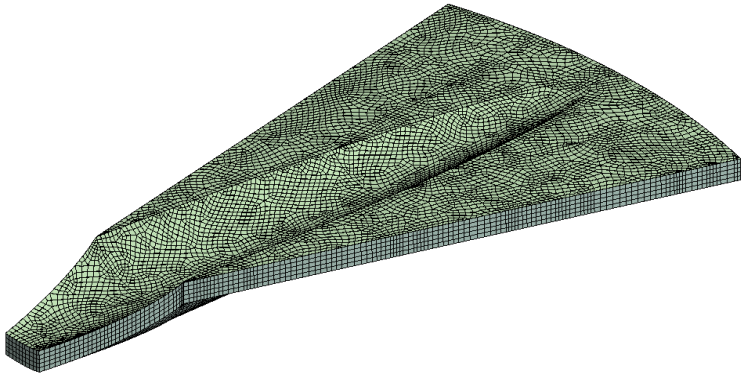




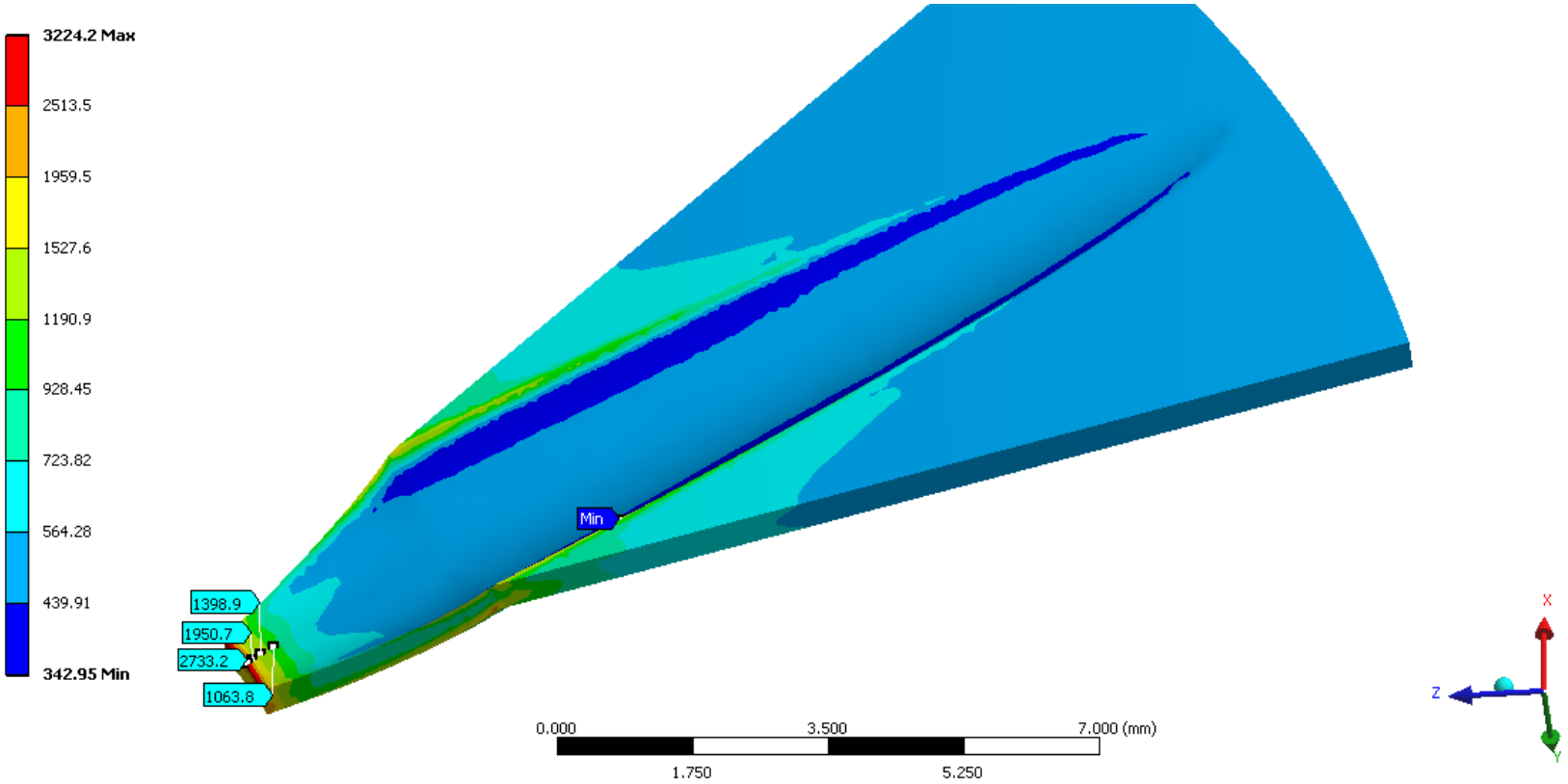
Geometric model adopted for FEM simulation test



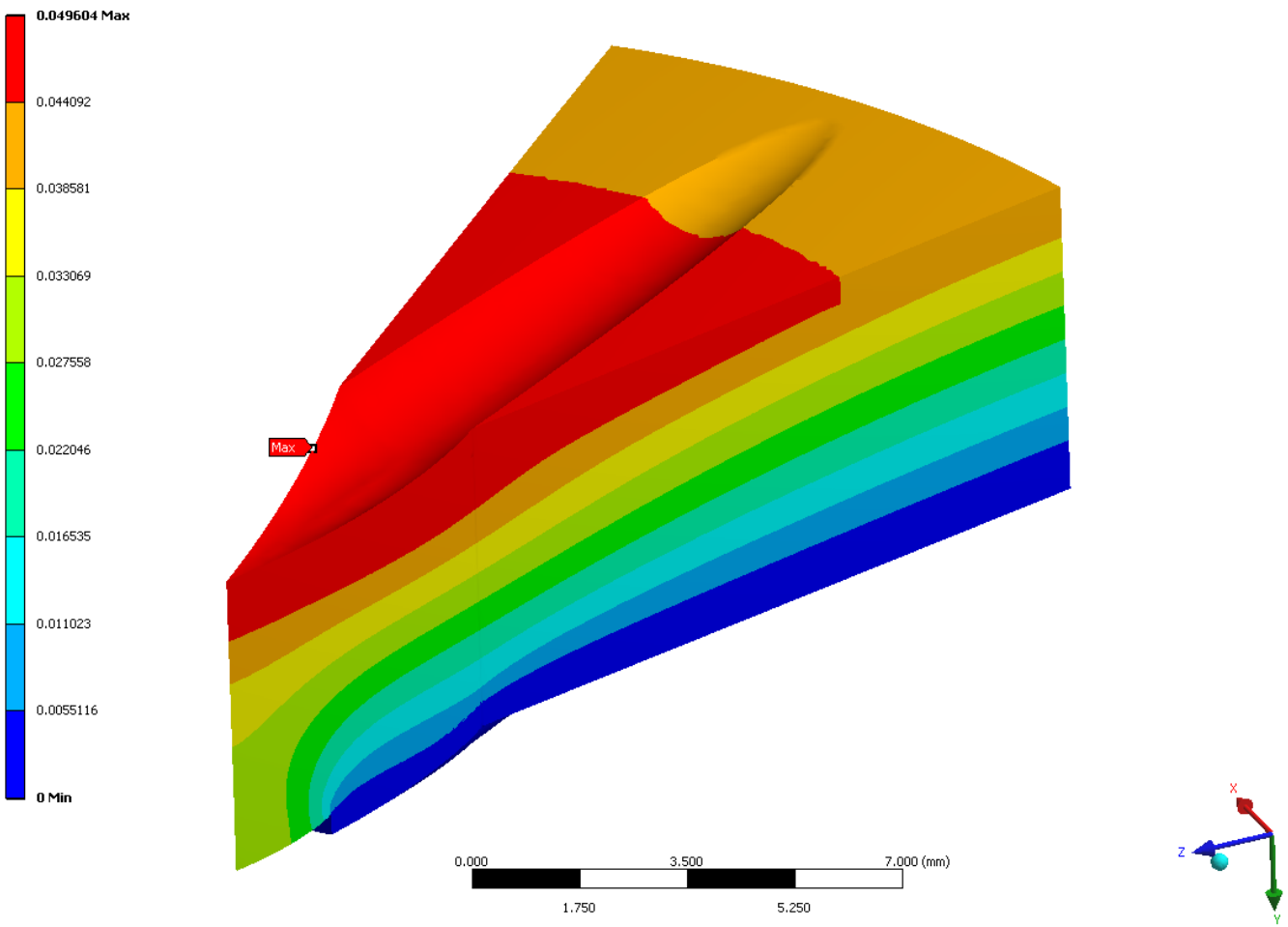




FEM numerical model



von Mises stresses in the coating



Displacement model

Summary

1. The morphology of the fracture of coatings is characterized with a dense structure, in some cases there is a columnar structure.

2. Hardness of the coatings tested fits within the range from 24 to 40 GPa.

3. The coatings are built of fine crystallites. Their size is 3÷13 nm.

4. On the basis of the scratch test, it was found that the critical load L_{C2} fitted within the range 46÷54 N .

5. The corrosion current density for the coatings tested fits within the range 0.15÷0.77 μAcm^{-2}, which proves their good anti-corrosion properties.

6. The tests made with the use of GDOS indicate the occurrence of a transition zone between the substrate material and the coating, which affects the improved adhesion between the coatings and the substrate.

7. Under the technically dry friction conditions, the friction coefficient for the associations tested is within the range 0.03-0.06 for all the investigated coatings.

8. Wear induced via extrusion tests showed a good durability of the dies coated with the investigation coatings.

9. Basing on the computer simulation of stresses developed during extrusion process in the CrN+DLC coating deposited on the die from the X40CrMoV5-1 hot work tool steel in the PVD process, the stresses were revealed. Stresses values are in the range 350 ÷ 2700 MPa.

Future research

In order to evaluate with more detail the possibility of applying these surface layers in tools, further investigations should be concentrated on the determination of the thermal fatigue of the coatings.

Thank you very much for your kind attention