Forming of the surface structure and properties of tool‘s ceramic inserts with improved abrasion resistance

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Aim of the work

A scientific objective of this work is to examine the structure and properties and to recognise the wear mechanisms of newly developed coatings applied in physical vapour deposition (PVD) processes with cathodic arc evaporation (CAE) techniques, as well as with chemical vapour deposition in a high-temperature CVD process on tools produced from ceramic sintered tool materials, with the intention to improve cutting properties of the investigated materials by considerably improving the life of the tools' cutting edge.

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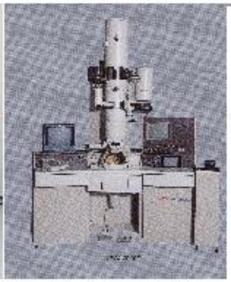
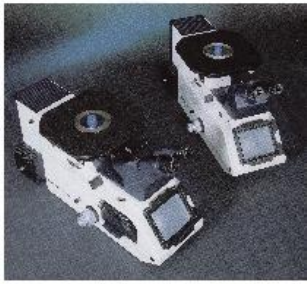
Researches methodology

State-of-the-art materials science research methods were employed for explaining the causes of a marked improvement in the operational properties of tools, especially thin foils tests, including on cross sections in a high-voltage transmission electron microscope.

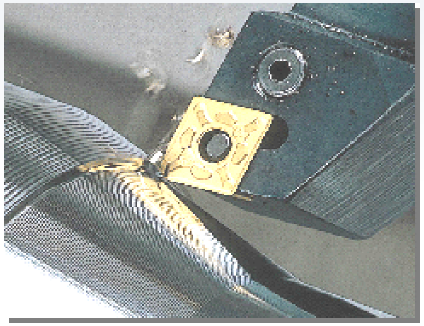







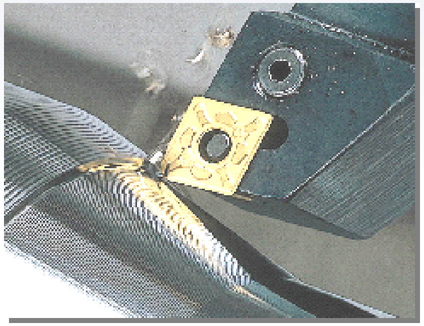
Researches methodology

- **Examinations of the structure:**
  - ✓The Scanning Electron Microscopy (SEM);
  - ✓The Transmission Electron Microscopy (TEM).
- **The study of chemical composition and phase:**
  - ✓X-ray of qualitative and quantitative microanalysis;
  - ✓Optical emission spectrometry (GDOS);
  - ✓X-ray qualitative phase analysis;
  - ✓Texture analysis;

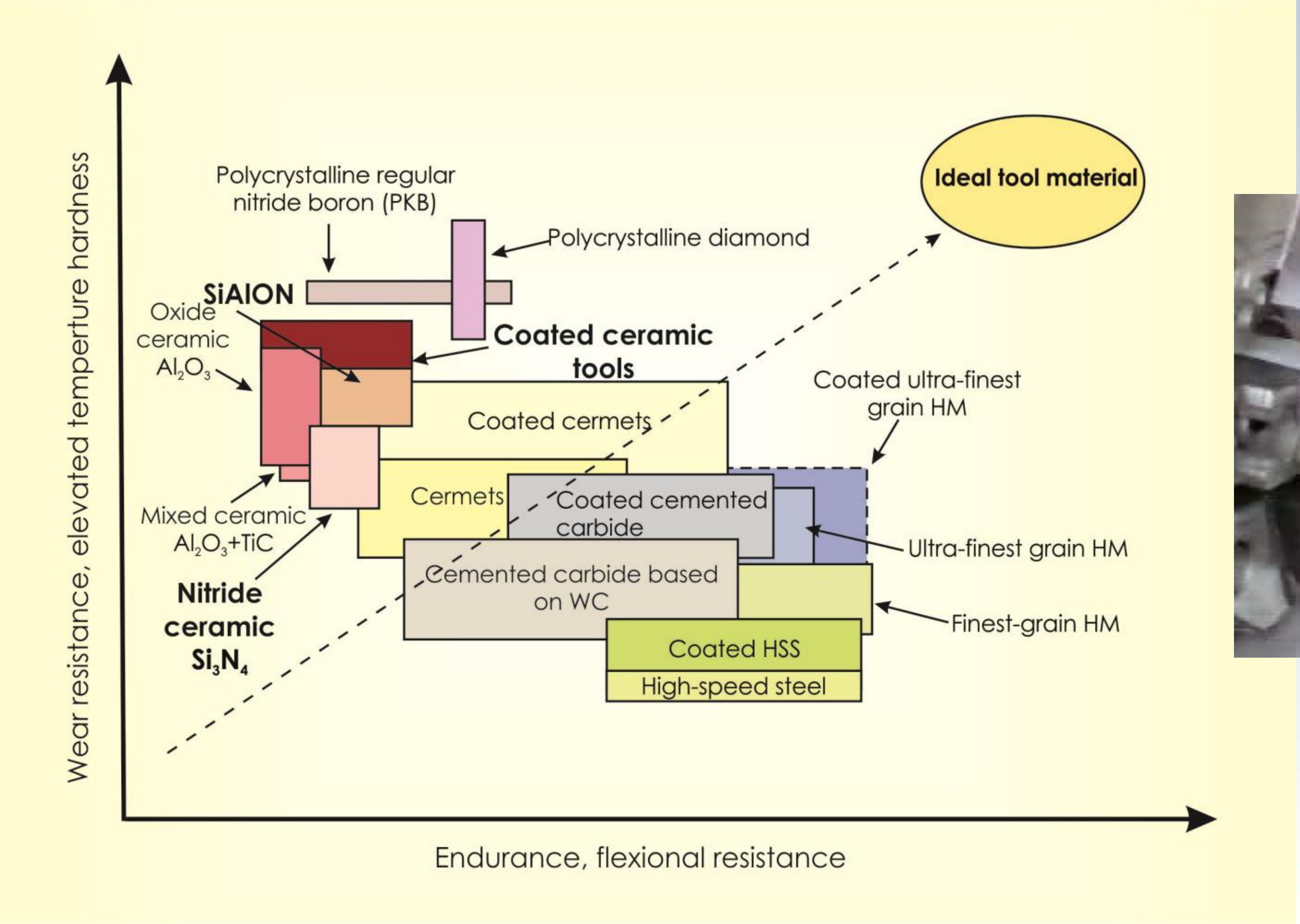


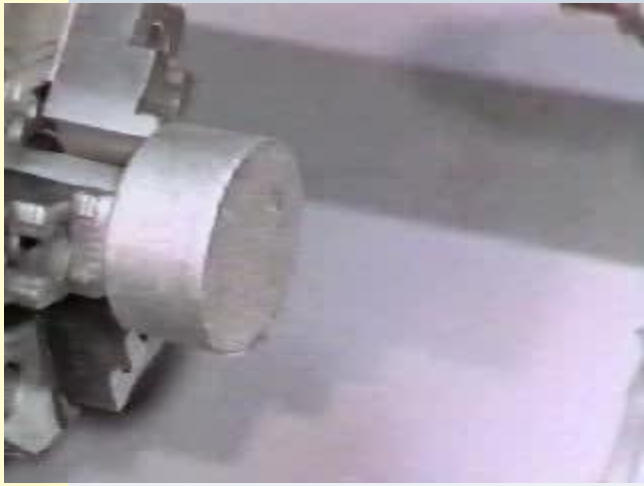
Researches methodology

- **Research of the mechanical properties:**
  - ✓Roughness;
  - ✓Dynamic hardness;
  - ✓Internal stresses;
  - ✓Adhesion - „scratch test”;
- **Research of the exploitative properties :**
  - ✓Wear resistance – „pin-on-disc”;
  - ✓Try cutting cast iron

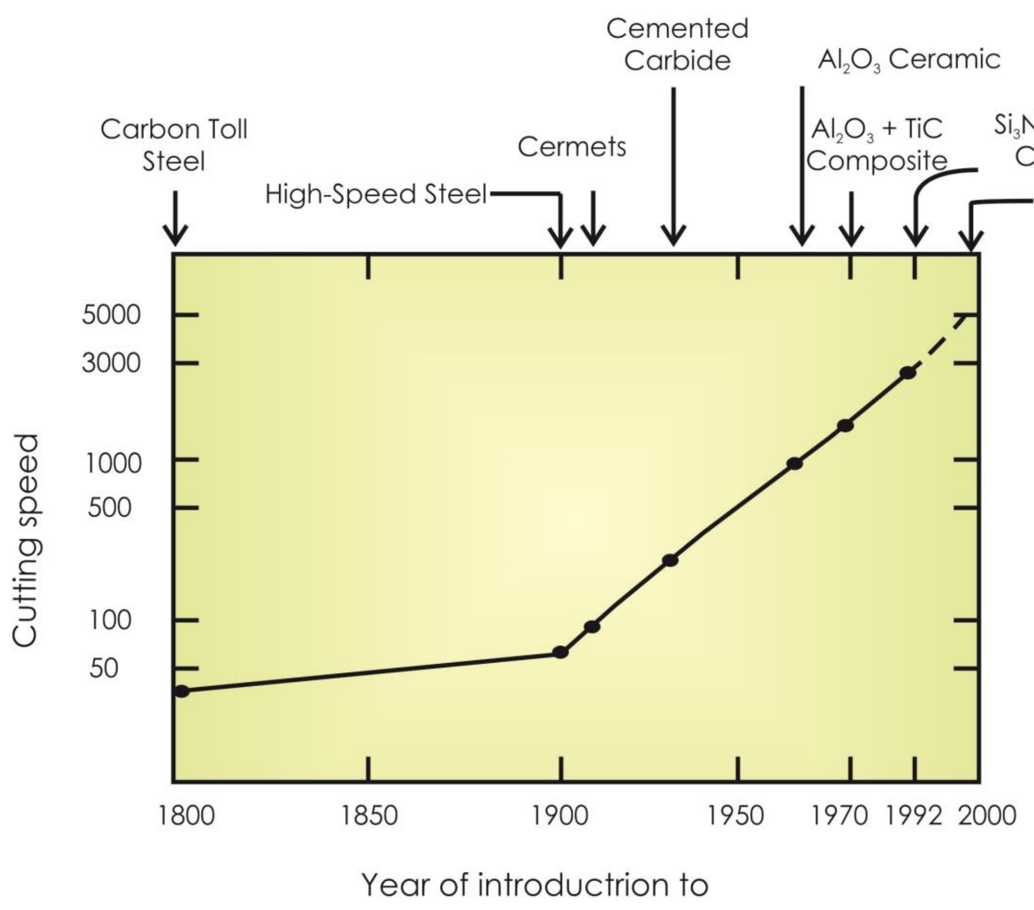


Ideal tool material





Ideal tool material



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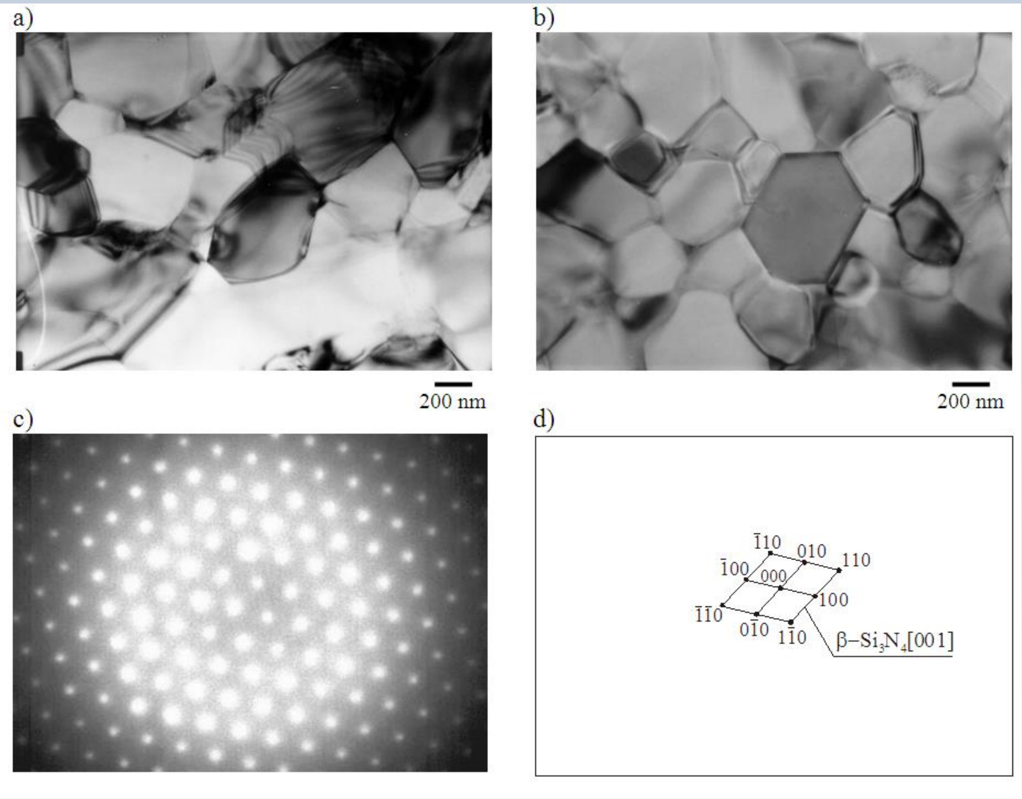
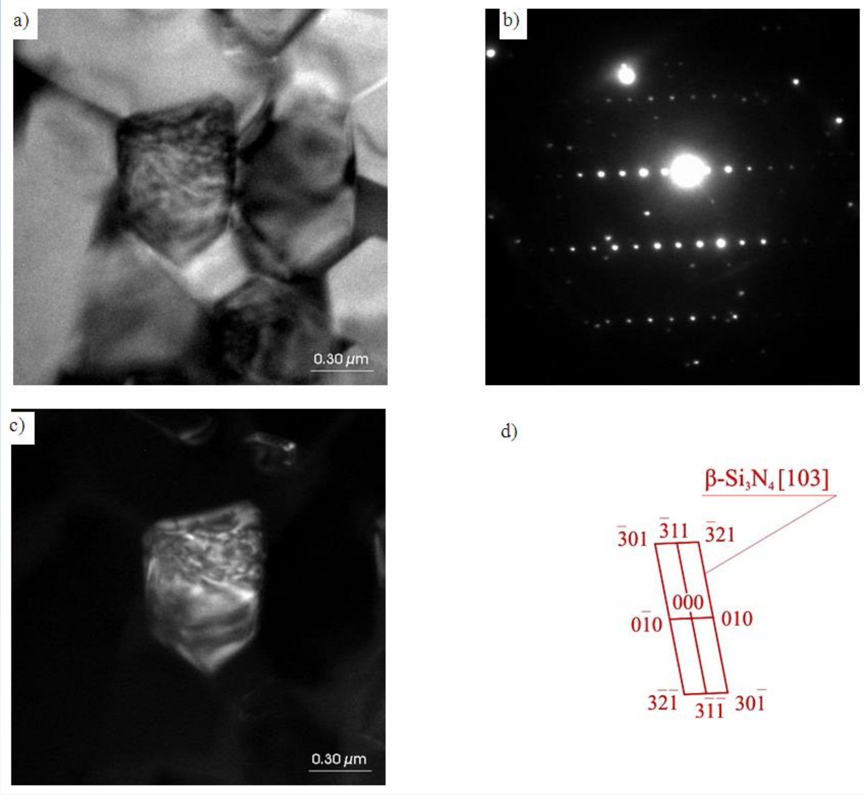
Structure of ceramics tool

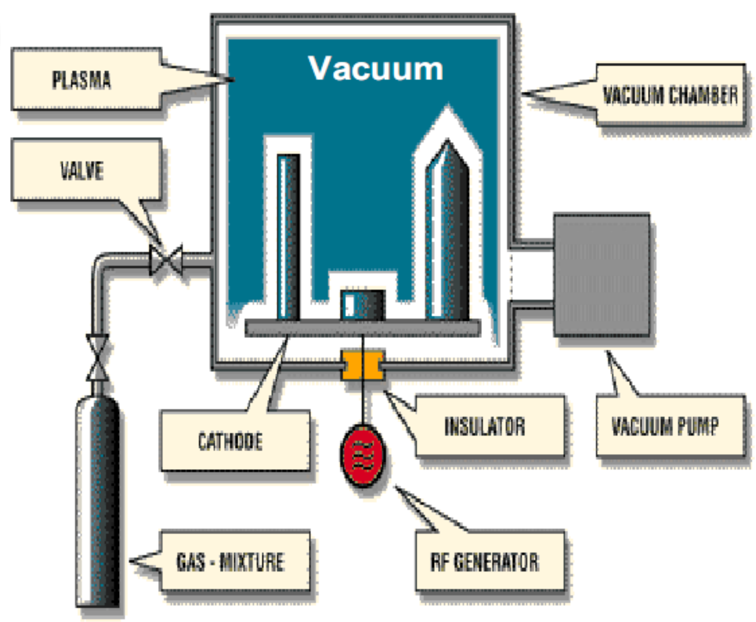




Si_3N_4

SiAlON

Chemical Vapour Deposition (CVD)



Source: Gas or Liquid

Conformal deposition (higher pressure)

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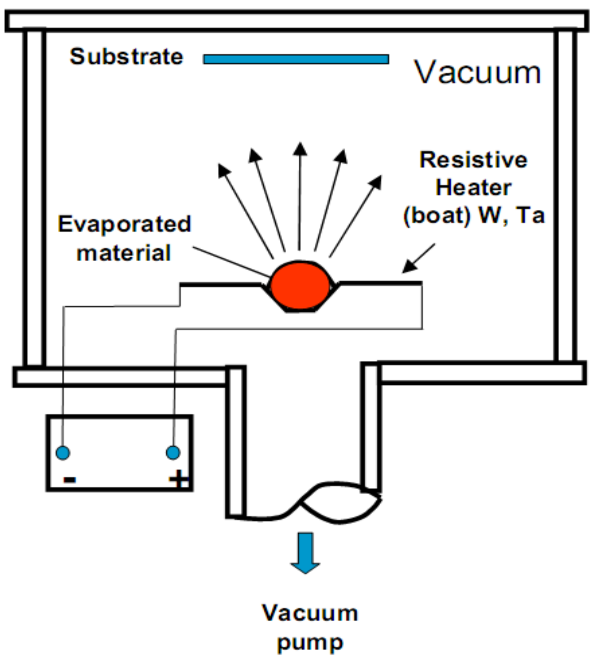
Physical Vapour Deposition (PVD)



- Evaporation involves two basic processes: a hot source material evaporates and condenses on the substrate.

Source: Solid

Directional ("line of sight") deposition

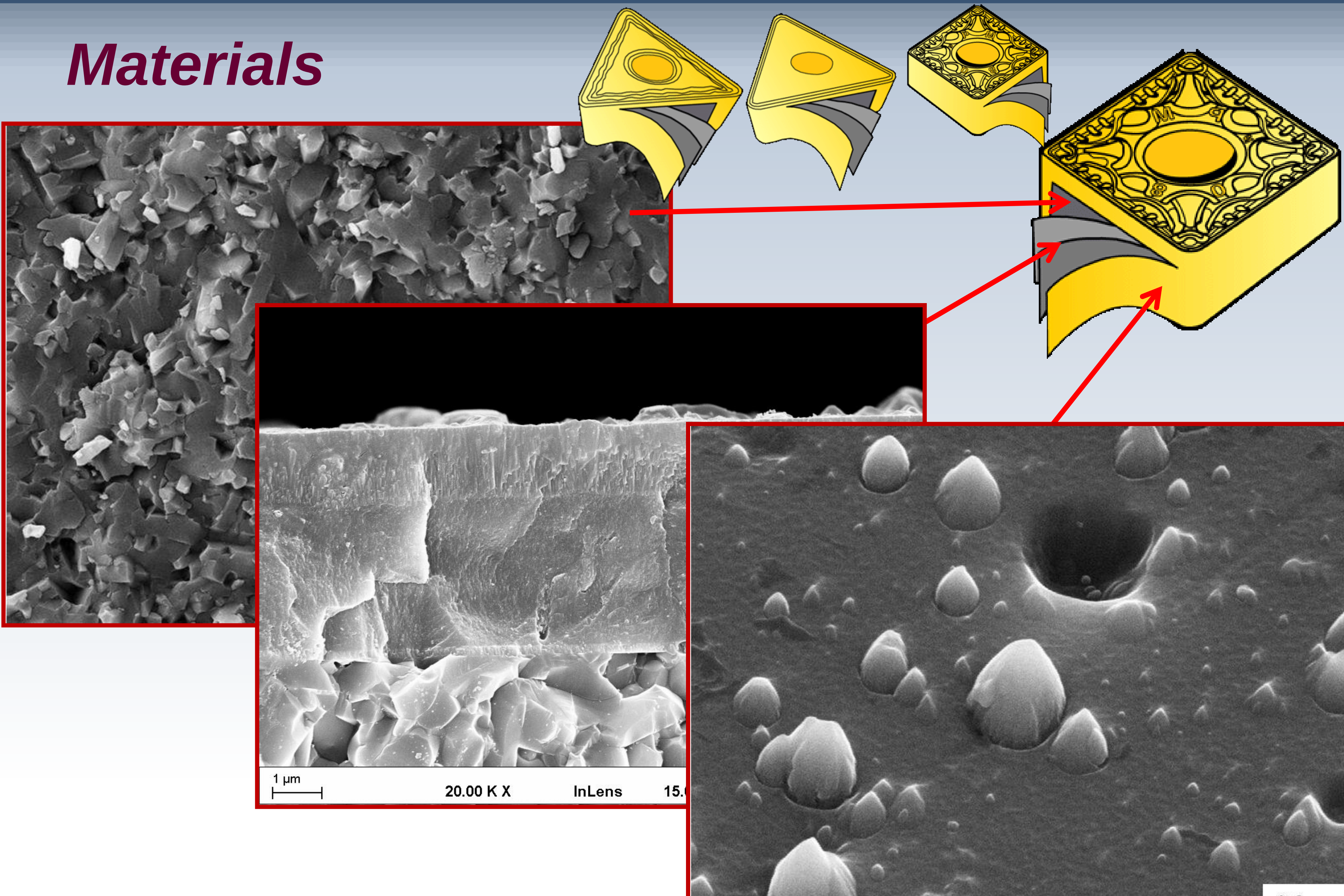
Chemical Vapour Deposition (CVD) *Physical Vapour Deposition (PVD)*

	CVD	PVD
Deposition	• T = 800 - 1000°C	• T = 70 - 500°C
Cycle Time	• 8 – 24 hours	• 3-4 hours for cutting tools
Properties	• excellent adherence • coating thickness up to 20 μm	• excellent adherence • uniform coating thickness • typical coating thickness 3-5 μm
Applications	• cutting tools, forming tools	• cutting tools, forming tools, components, medical devices, decorative

combinations of: TiN, TiC, Ti(C,N) and Al_2O_3 phases

combinations of: TiN, Ti(B,N), (Ti,Zr)N, Ti(C,N), Ti(C,N)+(Ti,Al)N, (Ti,Al,Si)N (Al,Si,Ti)N, (Al,Ti)N, (Ti,Al)N, (Al,Cr)N phases

Materials



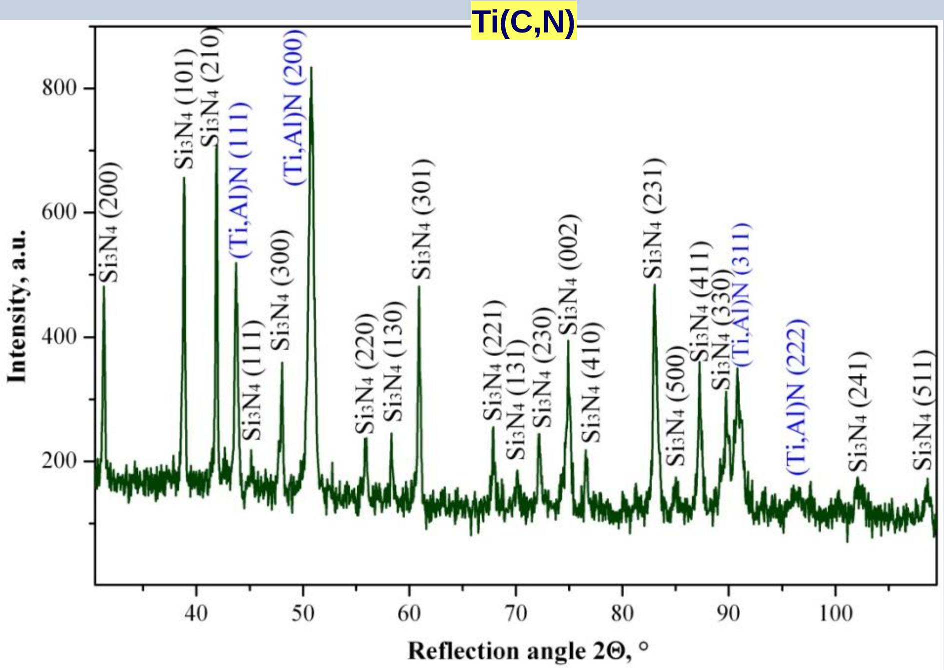
Materials

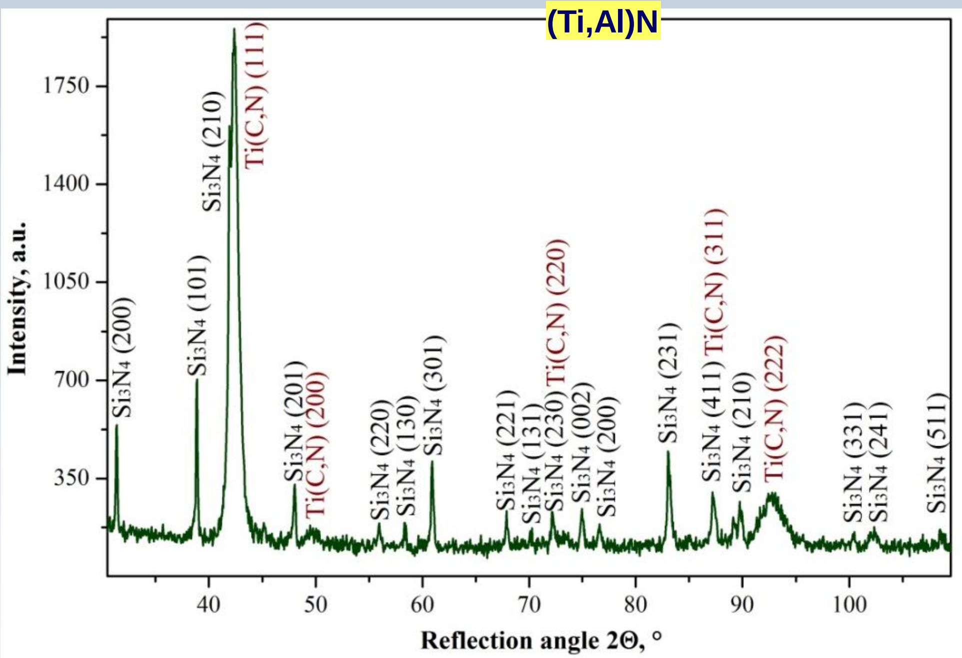
Process	Coating	Substrate	Thickness, µm
PVD	TiN	Si_3N_4	0.8
	TiN+multi(Ti,Al,Si)N+TiN	Si_3N_4	4.0
	TiN+(Ti,Al,Si)N+TiN	Si_3N_4	2.0
	TiN+(Ti,Al,Si)N+(Al,Si,Ti)N	Si_3N_4	2.5
	Ti(B,N)	Si_3N_4	2,1
	Ti(C,N)	Si_3N_4	2,1
	(Ti,Zr)N	Si_3N_4	1,7
	(Ti,Al)N	Si_3N_4	0,7
	(Ti,Al)N+(Al,Cr)N	Sialon ceramics	4,0
	(Al,Cr)N+(Ti,Al)N	Sialon ceramics	3,9
	Ti(C,N)+(Ti,Al)N	Sialon ceramics	1,4
	(Al,Ti)N	Sialon ceramics	3,0
	(Ti,Al)N	Sialon ceramics	5,0
	(Al,Cr)N	Sialon ceramics	4,8
	Ti(C,N) (2)	Sialon ceramics	1,8
	Ti(C,N) (1)	Sialon ceramics	1,5
	Ti(B,N)	Sialon ceramics	1,3
	(Ti,Zr)N	Sialon ceramics	2,3
CVD	Ti(C,N)+TiN	Si_3N_4	4.2
	Ti(C,N)+Al_2O_3+TiN	Si_3N_4	9.5
	TiC+TiN	Si_3N_4	5.4
	TiC+Ti(C,N)+Al_2O_3+TiN	Si_3N_4	7.8
	TiN+Al_2O_3	Si_3N_4	10.0
	TiN+Al_2O_3+TiN*	Si_3N_4	3.8
	Al_2O_3+TiN (1)*	Si_3N_4	2.6
	Al_2O_3+TiN (2)*	Si_3N_4	1.7
	TiN+Al_2O_3+TiN+Al_2O_3+TiN*	Si_3N_4	4.5
	Ti(C,N)+ Al_2O_3+TiN	Sialon ceramics	7.0
	Ti(C,N)+ TiN	Sialon ceramics	2.8

* płytki skrawające różnych producentów

In total were tested about 30 coatings on these substrates

X-ray qualitative phase analysis

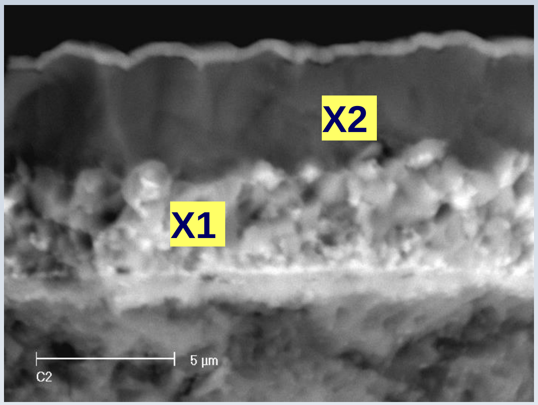


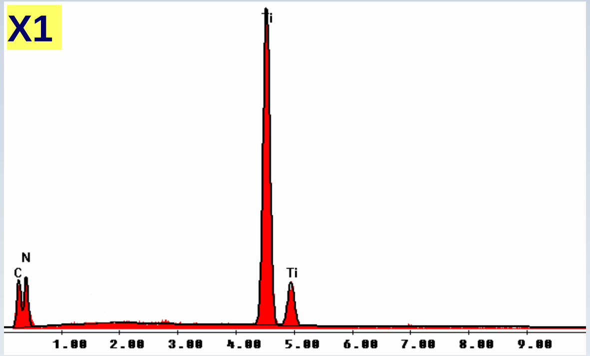


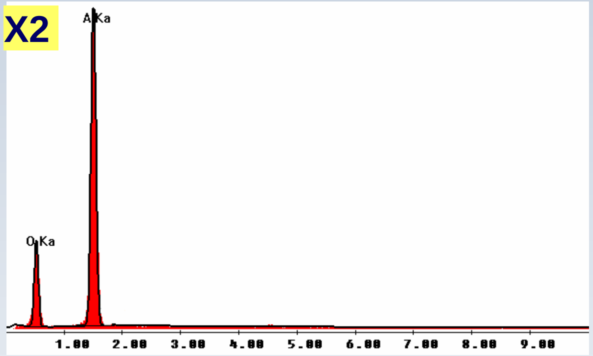
A qualitative phase composition analysis carried out with the X-ray diffraction method allows to conclude that, as assumed, coatings were formed on the tool ceramics substrates containing TiN, Ti(C,N), AlN and CrN phases and an Al_2O_3 phase in case of CVD coatings.

The chemical composition of the coatings

$Ti(C,N)+Al_2O_3+TiN$







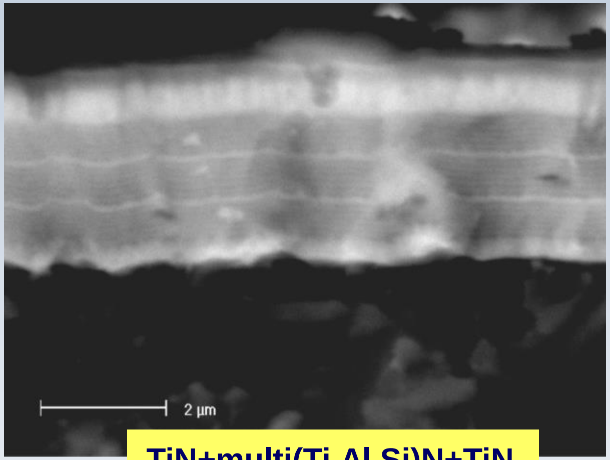
area X1

Element	Mass concentration of elements,%	Atomic concentration of elements, %
C	9.72	20.21
N	22.03	40.29
Ti	68.25	39.50
Sum	100	100

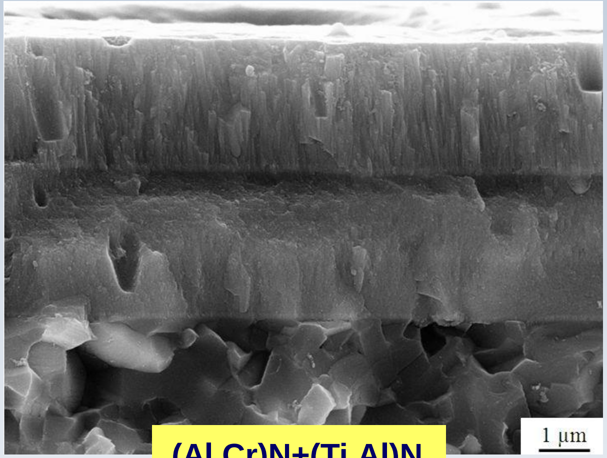
area X2

Element	Mass concentration of elements,%	Atomic concentration of elements, %
O	42.86	55.89
Al	57.14	44.11
Sum	100	100

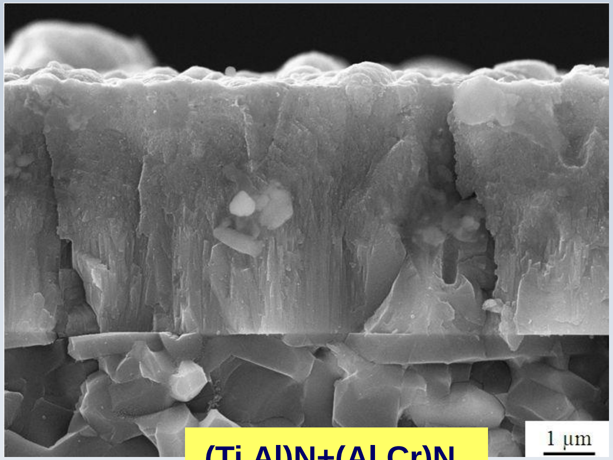
Structure of PVD coatings (SEM)



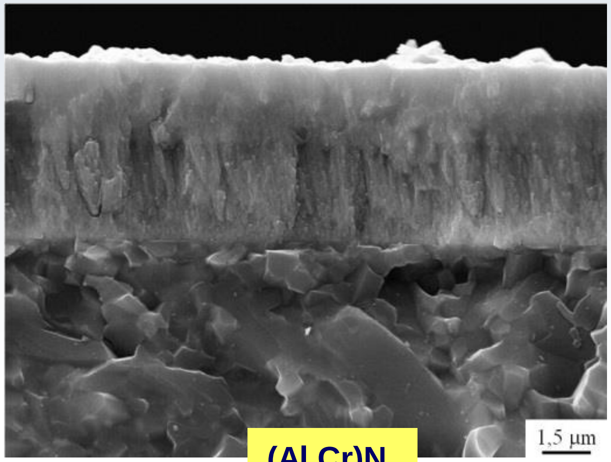
TiN+multi(Ti,Al,Si)N+TiN



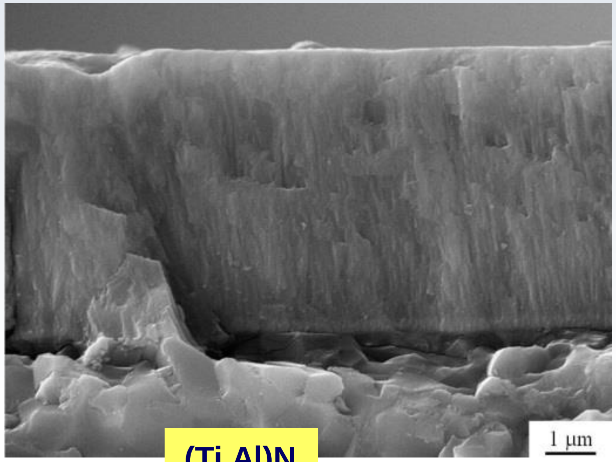
(Al,Cr)N+(Ti,Al)N



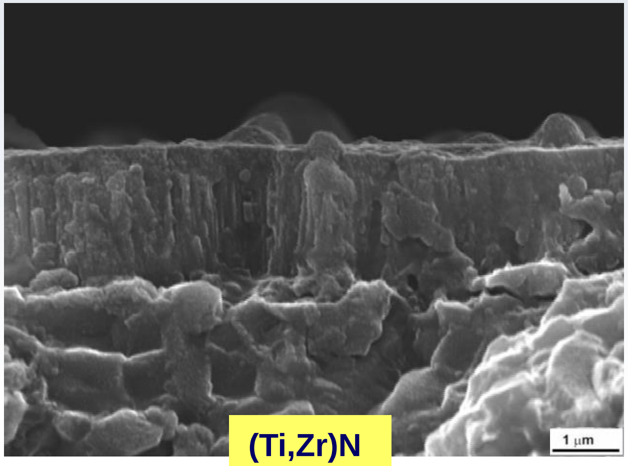
(Ti,Al)N+(Al,Cr)N



(Al,Cr)N

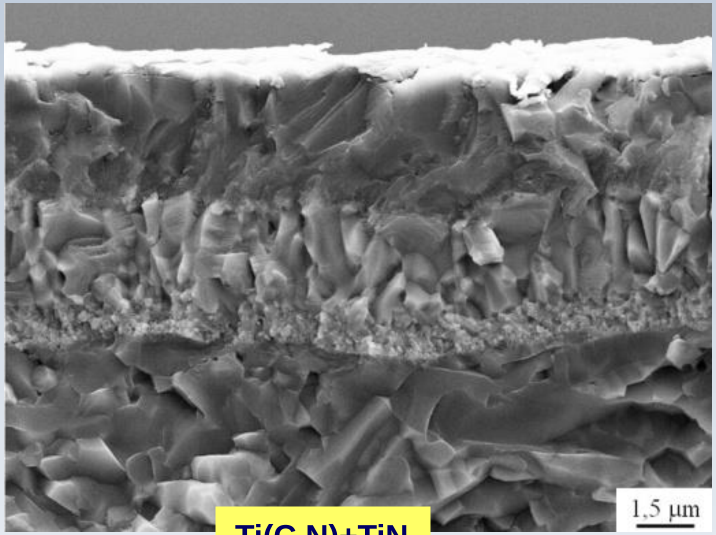


(Ti,Al)N

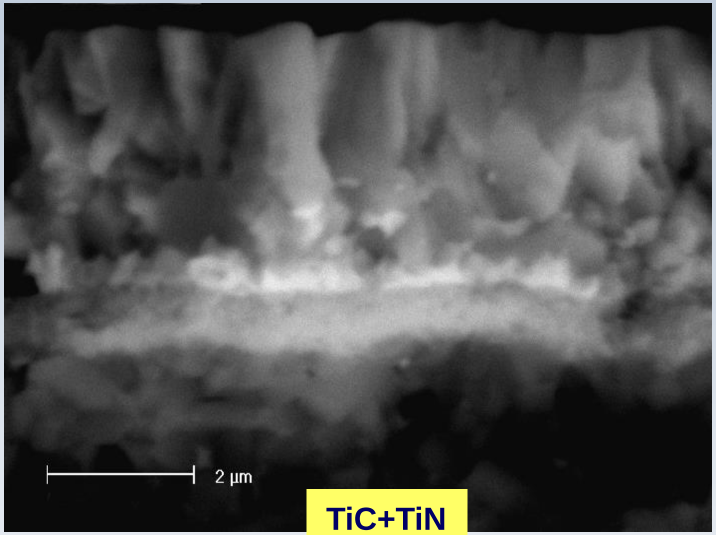


(Ti,Zr)N

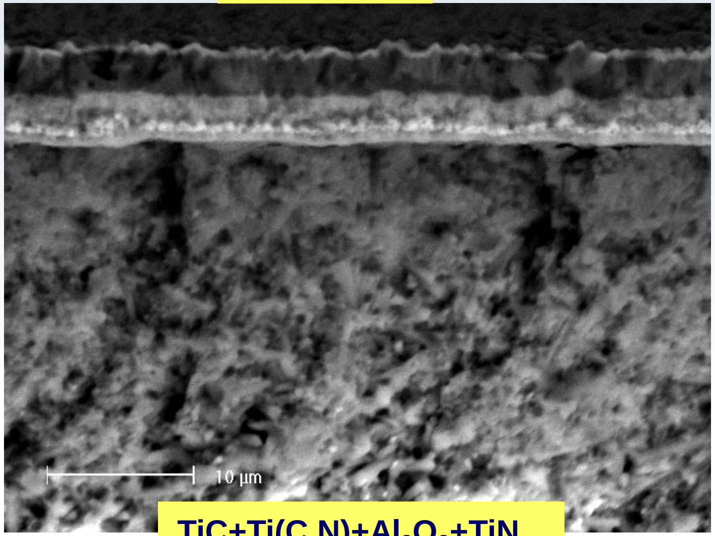
Structure of CVD coatings



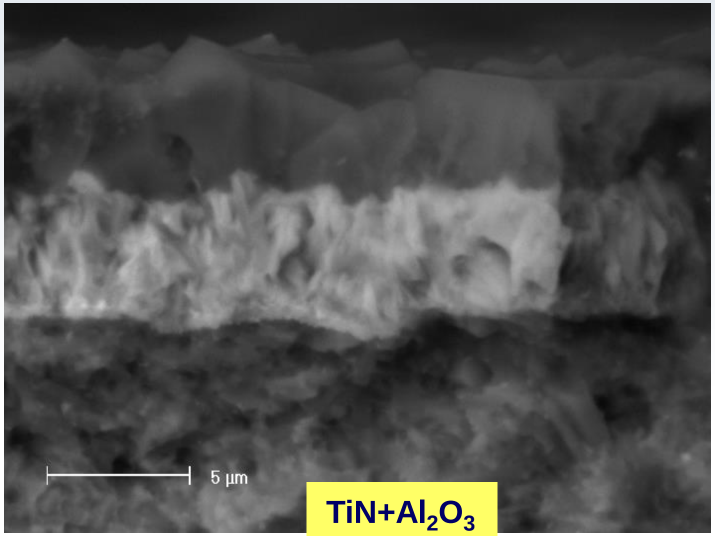
Ti(C,N)+TiN



TiC+TiN

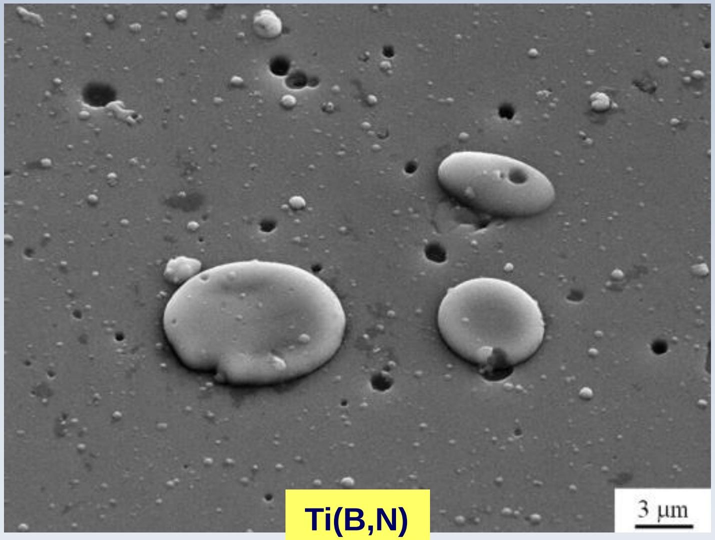


$TiC+Ti(C,N)+Al_2O_3+TiN$

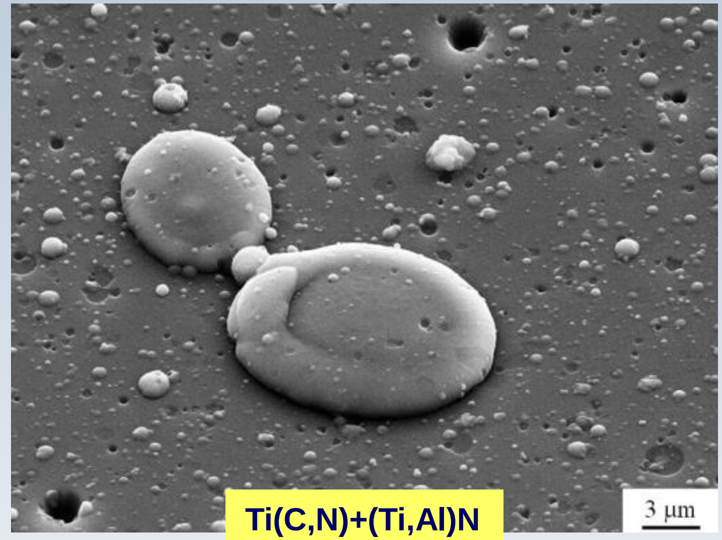


$TiN+Al_2O_3$

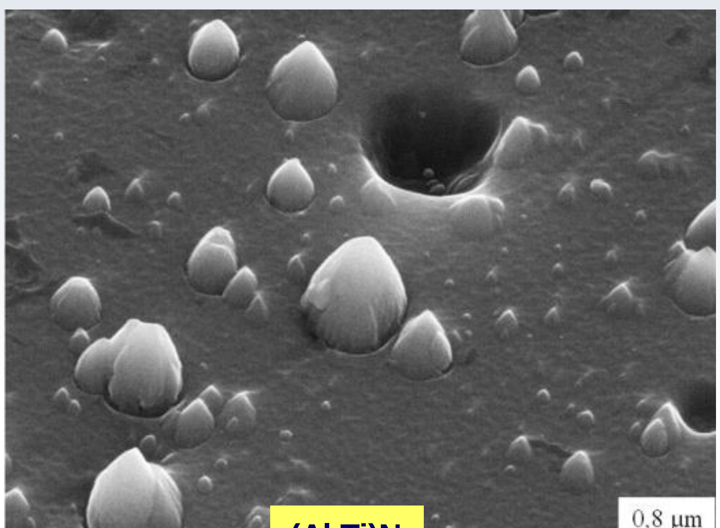
Surface topography of PVD coatings



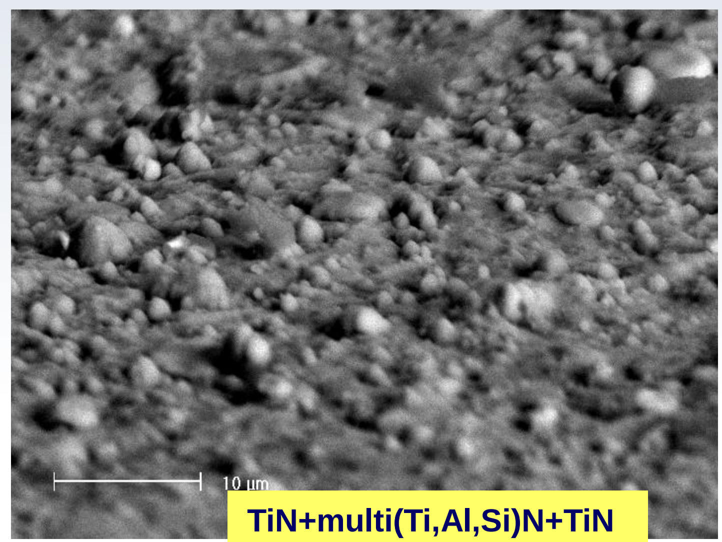
Ti(B,N)



Ti(C,N)+(Ti,Al)N

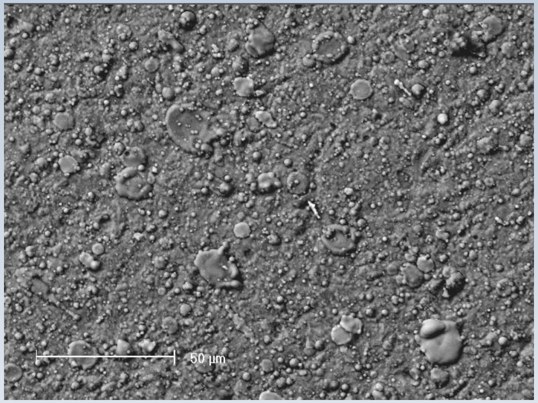


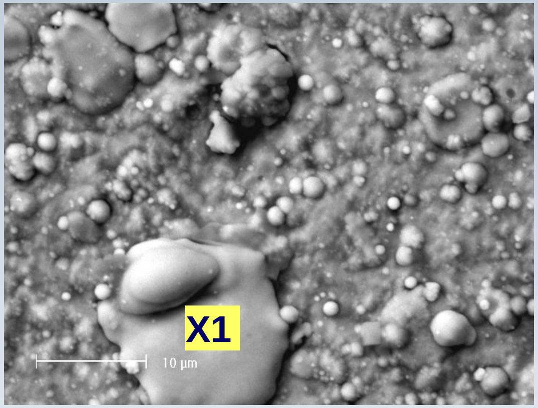
(Al,Ti)N

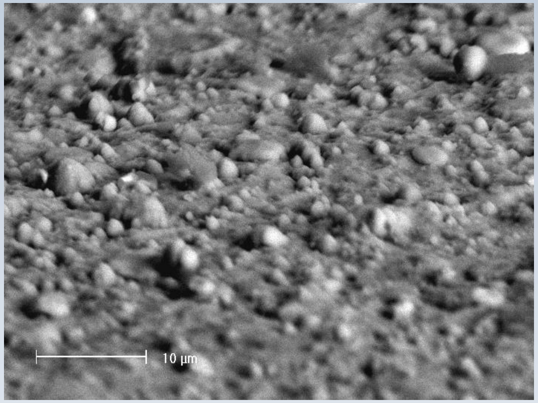


TiN+multi(Ti,Al,Si)N+TiN

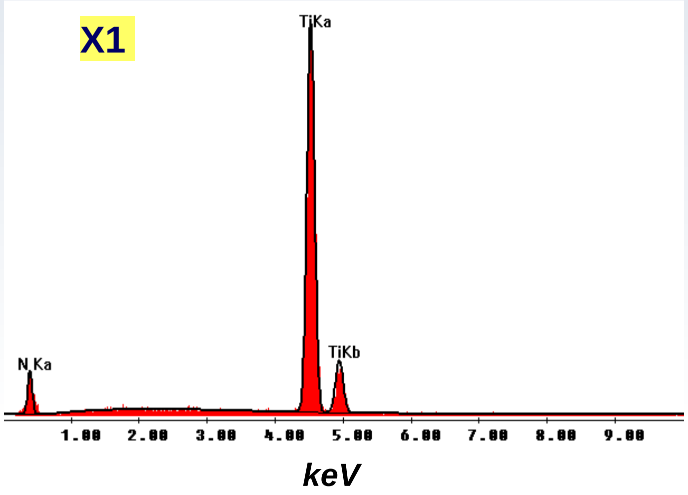
Surface topography of PVD coatings







TiN+multiTiAlSiN+TiN



area X1

Element	Mass concentration of elements,%	Atomic concentration of elements, %
N	16.24	39.86
Ti	83.76	60.14
Sum	100	100

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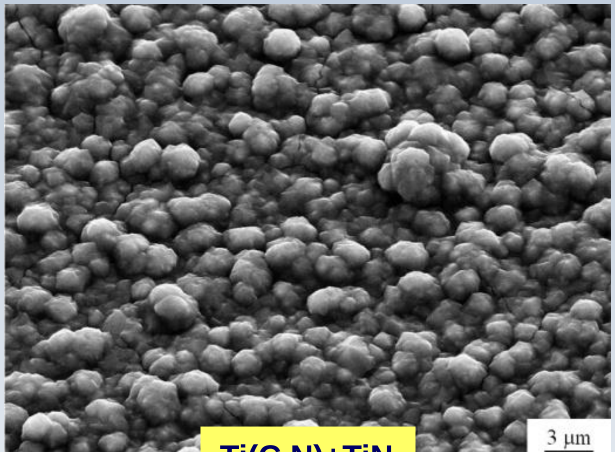
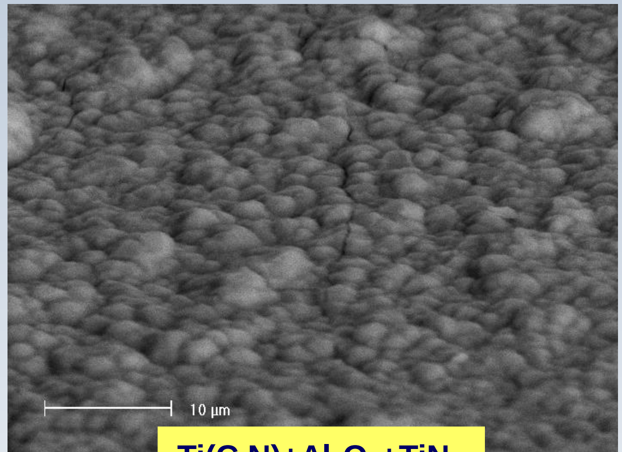
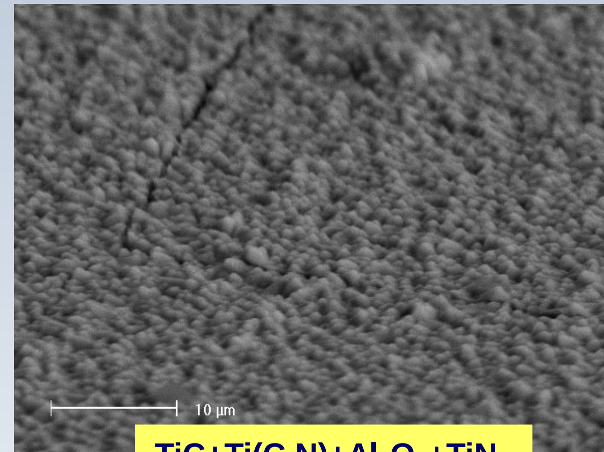
Surface topography of CVD coatings



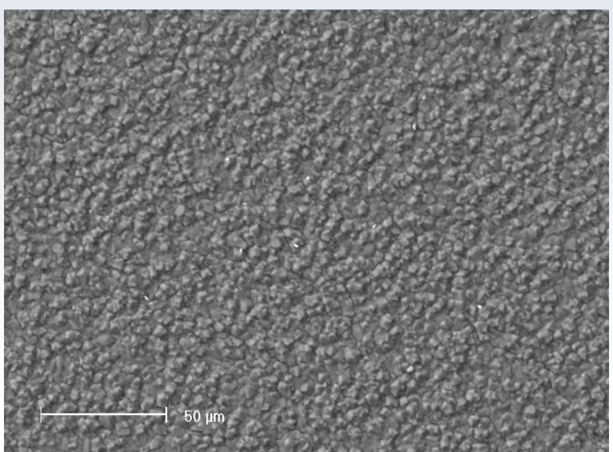


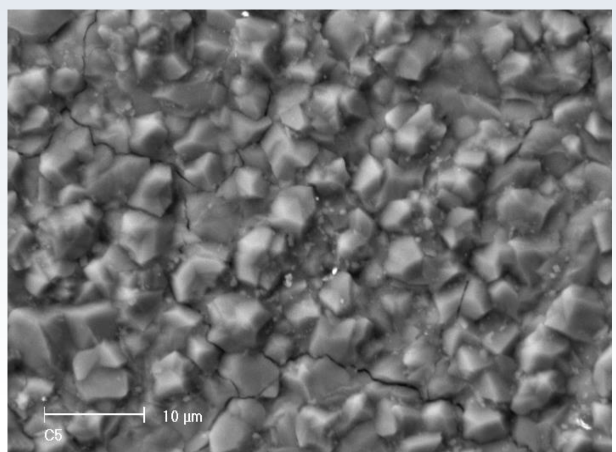


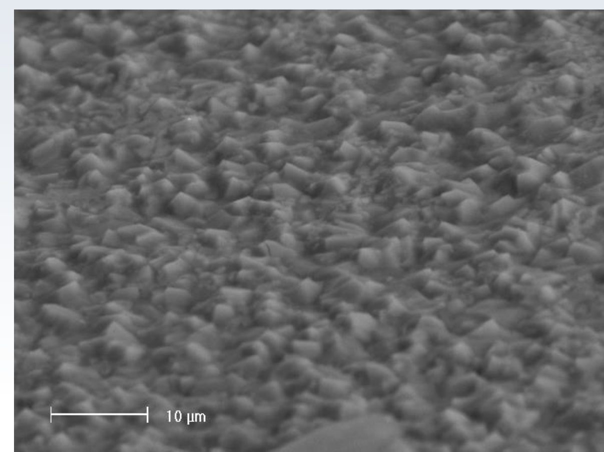
$Ti(C,N)+TiN$

$Ti(C,N)+Al_2O_3+TiN$

$TiC+Ti(C,N)+Al_2O_3+TiN$

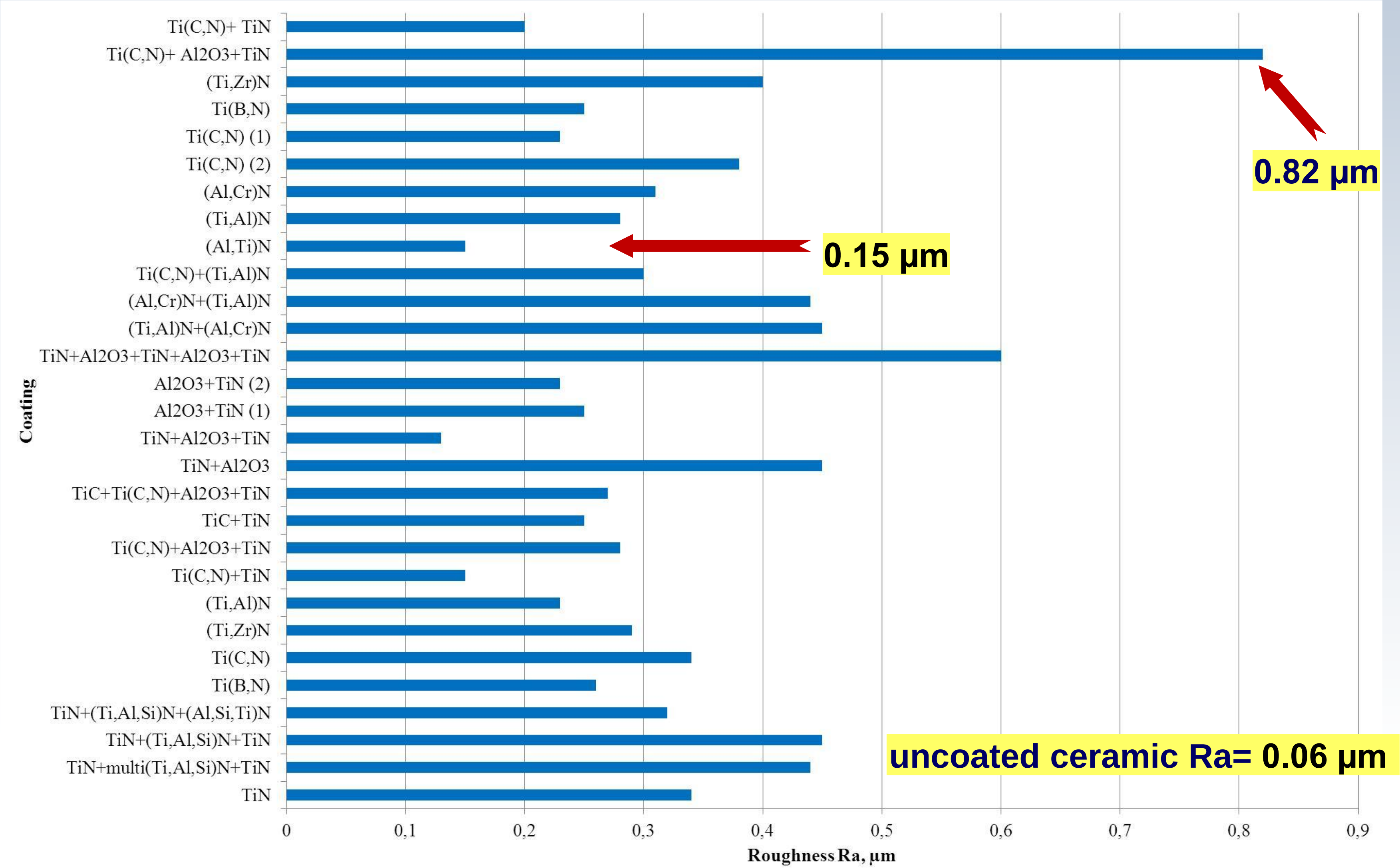






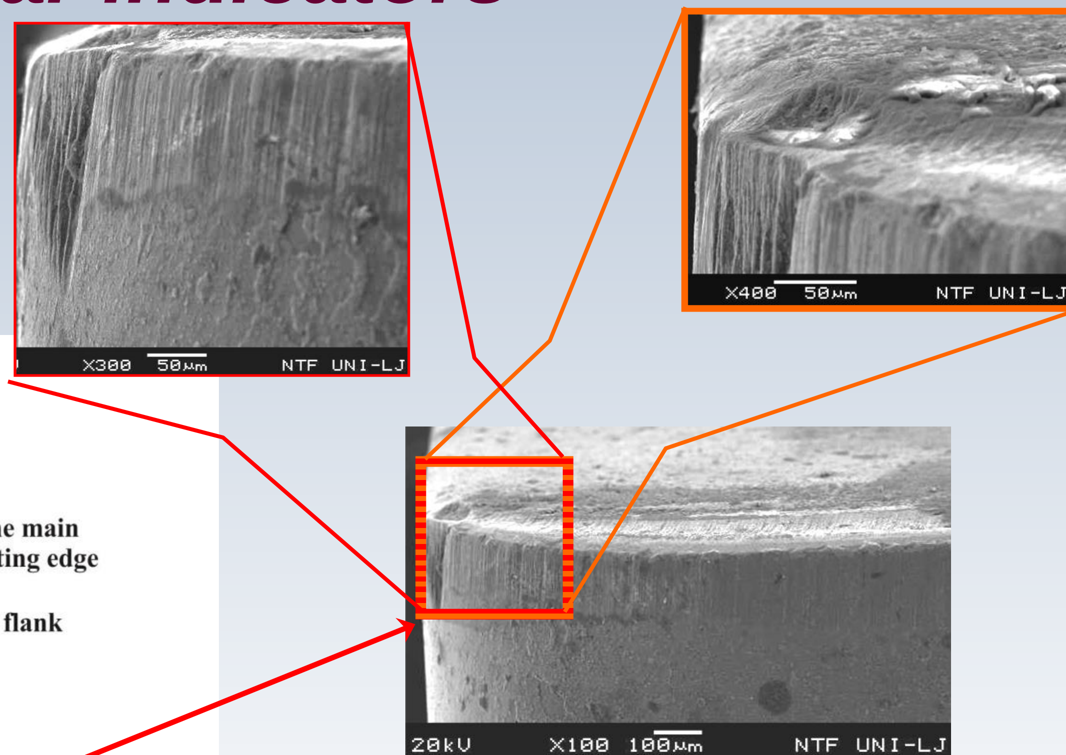
$TiN+Al_2O_3$

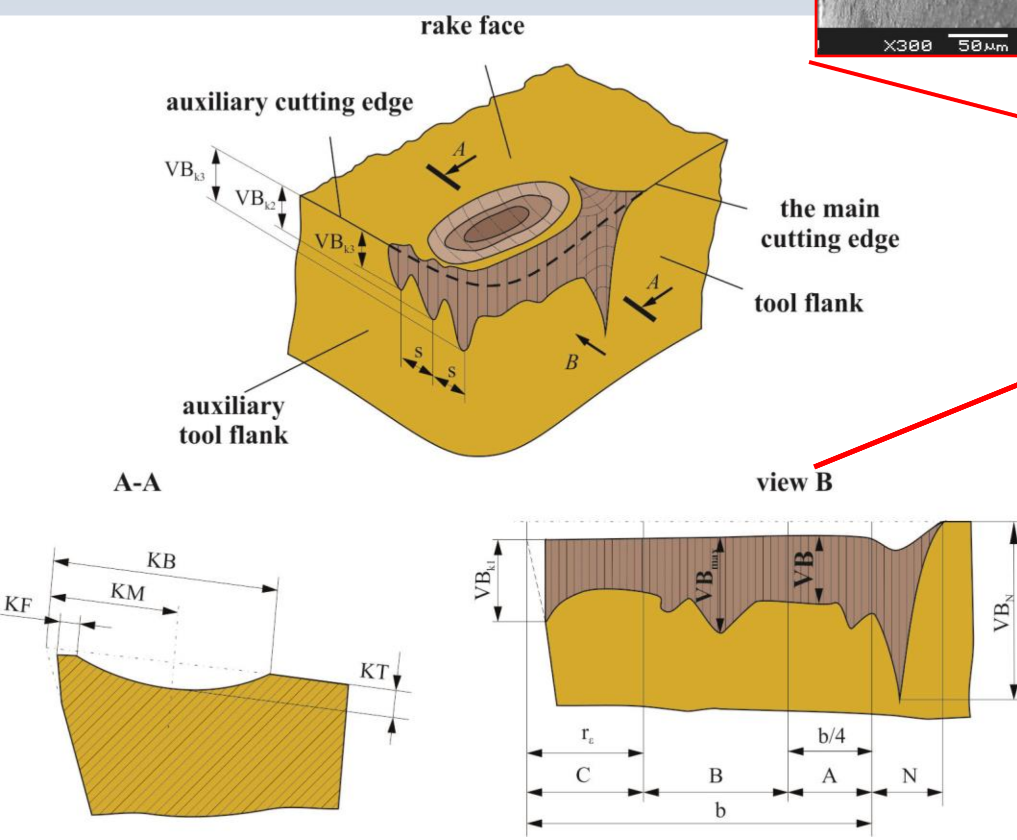
Coatings roughness



Cutting edges wear indicators

The cutting edge life is determined as the cutting time T expressed in minutes



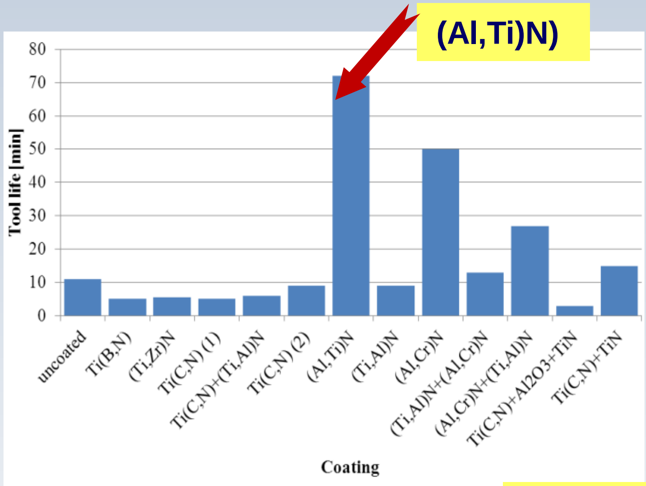


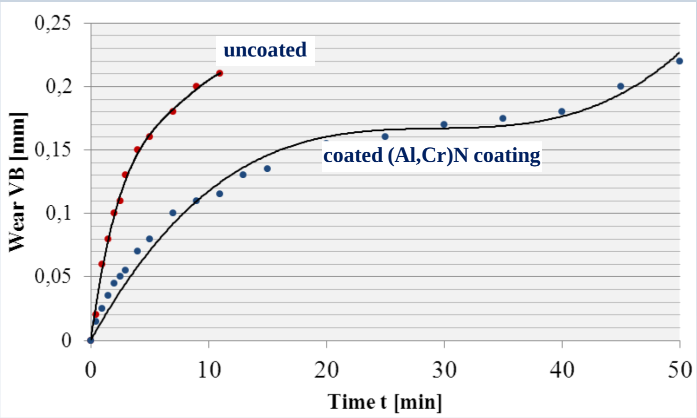
VB = 0.2 mm

v_c = 170 m/min

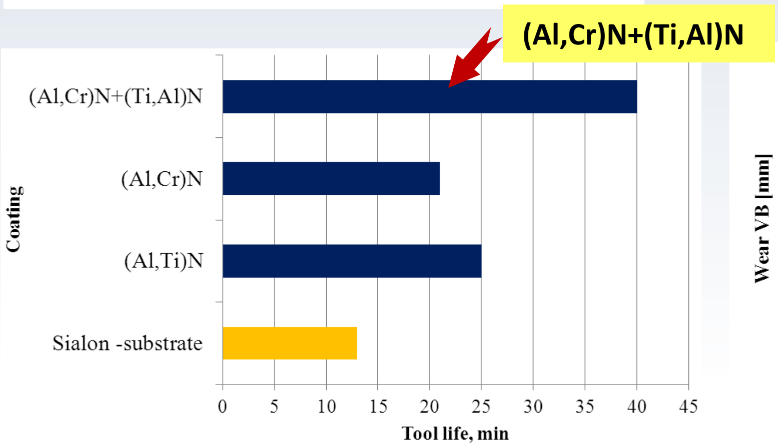
v_c = 425 m/min

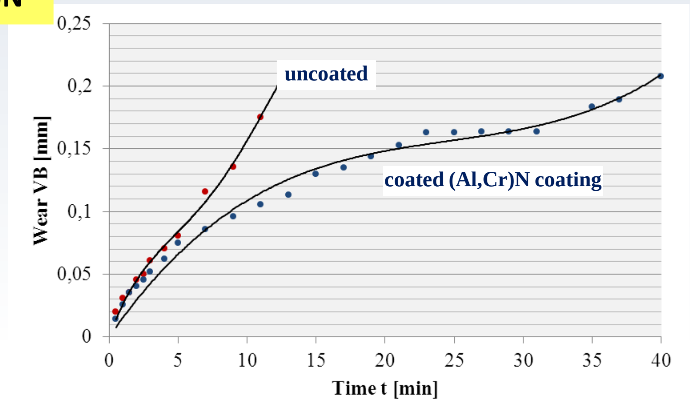
Cutting properties for coatings deposited on sialon ceramics





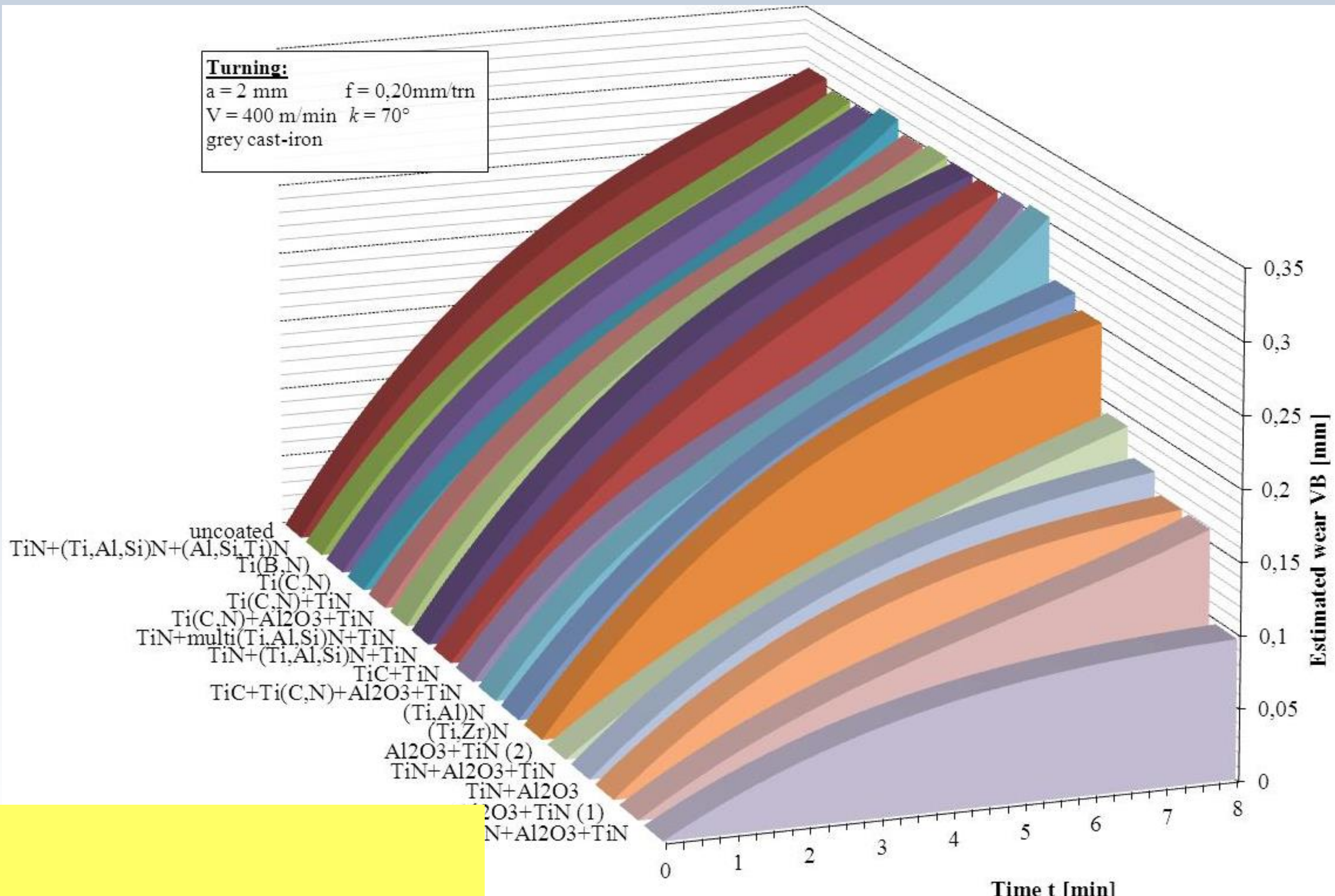
v_c = 170 mm/min
f = 0,2 mm/obr
a_p = 1 mm
VB = 0,2 mm





v_c = 425 mm/min
f = 0,2 mm/obr
a_p = 1 mm
VB = 0,2 mm

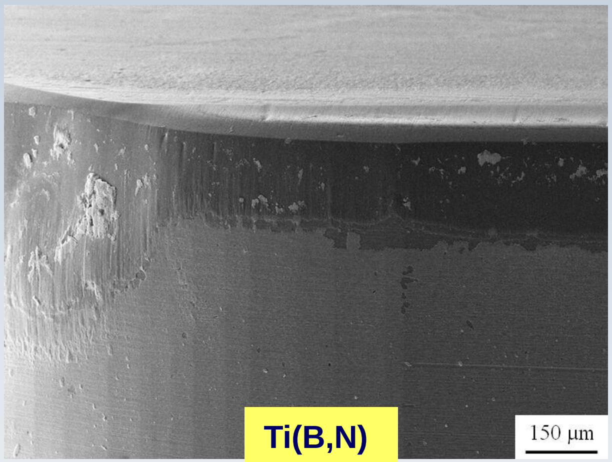
Cutting properties for coatings deposited on nitride ceramics



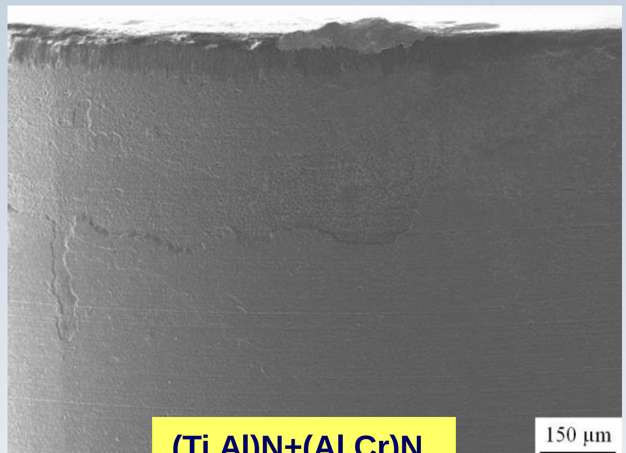
v_c = 400 mm/min
f = 0,2 mm/obr
a_p = 2 mm
VB = 0,3 mm

(Ti,Al)N
(Ti,Zr)N
coatings based Al_2O_3 and TiN phases

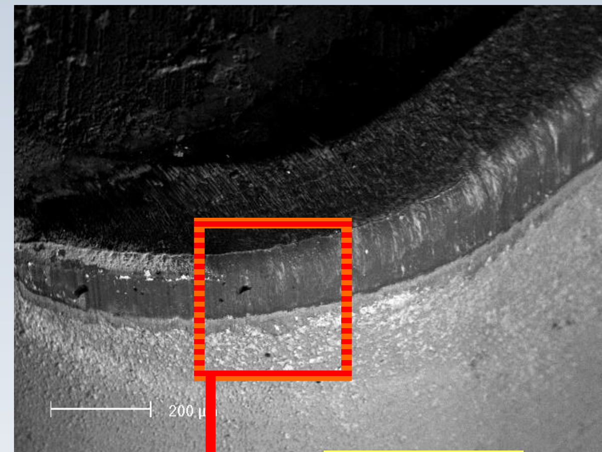
Cutting properties of tool ceramics with PVD and CVD coatings



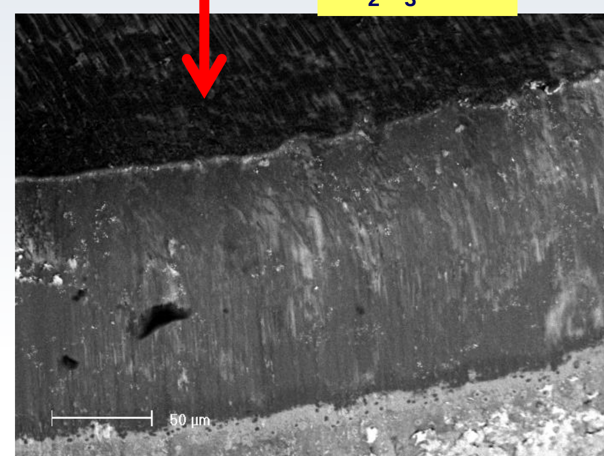
Ti(B,N)

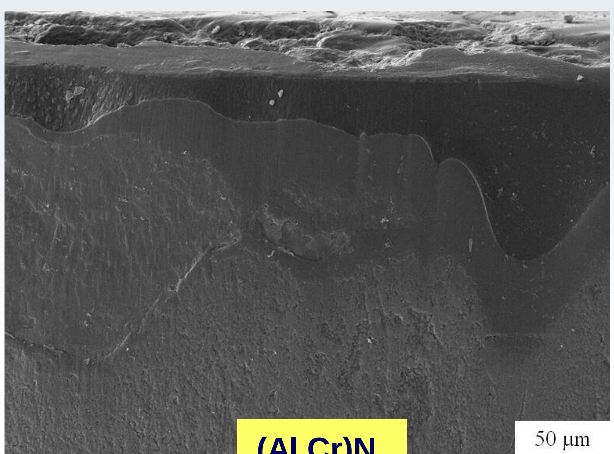


(Ti,Al)N+(Al,Cr)N

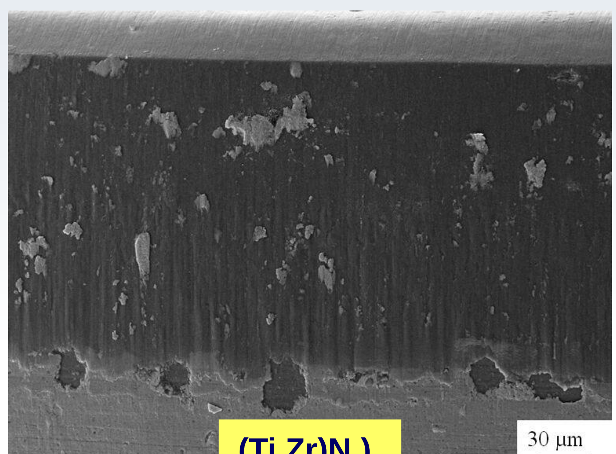


Al_2O_3+TiN



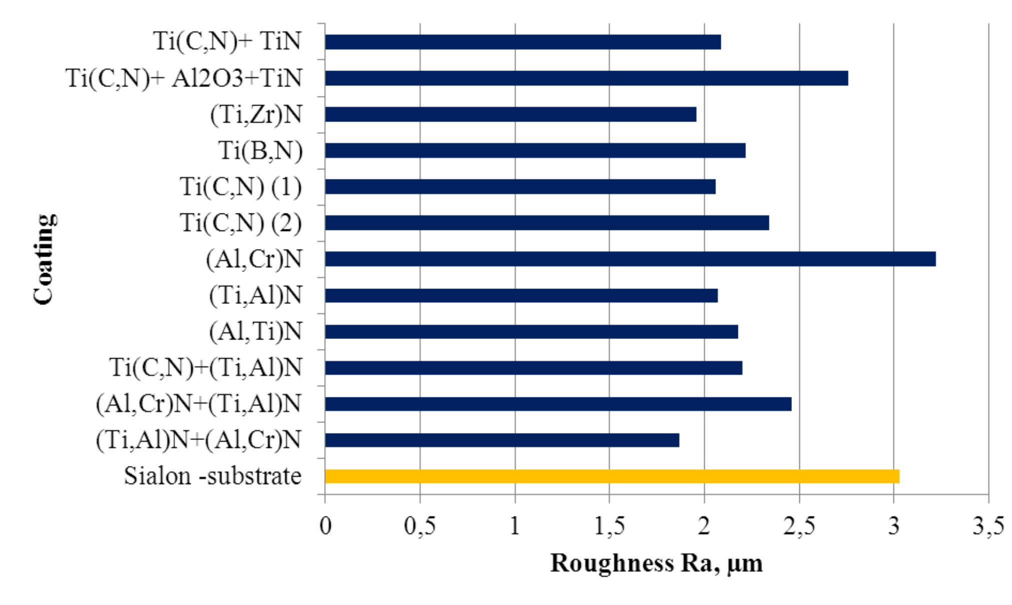


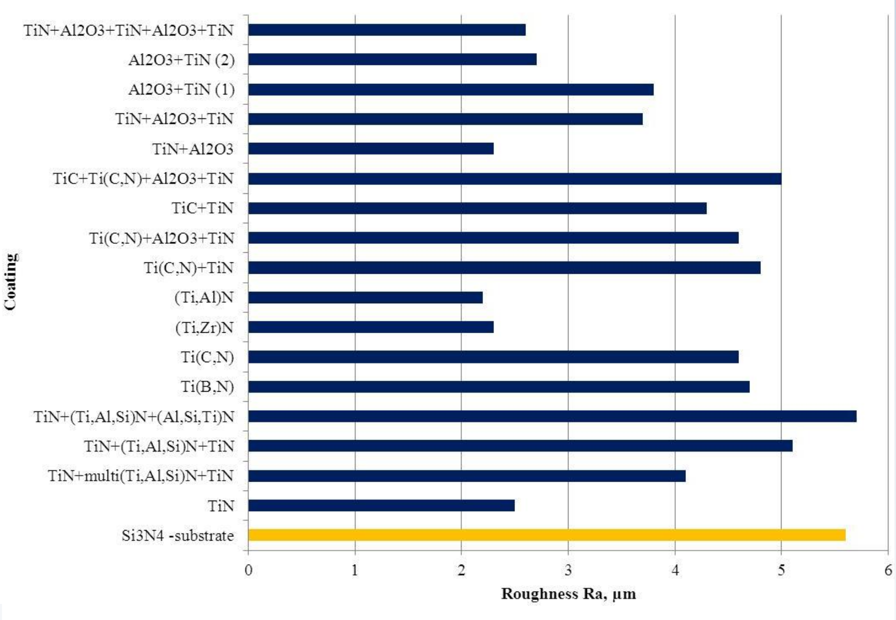
(Al,Cr)N



(Ti,Zr)N)

Cutting properties of tool ceramics with PVD and CVD coatings





All the tools coated with PVD and CVD coatings, for which improved cutting edge life was observed, contribute to a better quality of the machined surface

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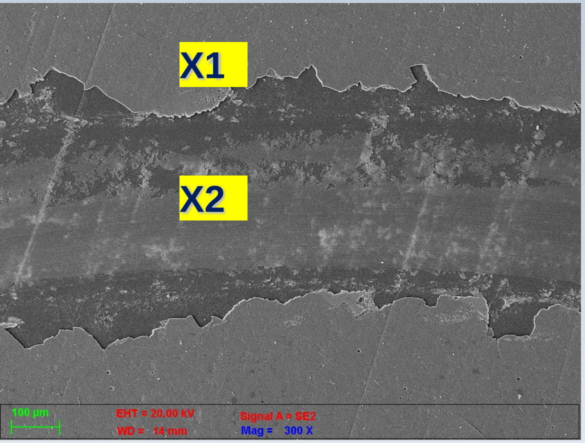
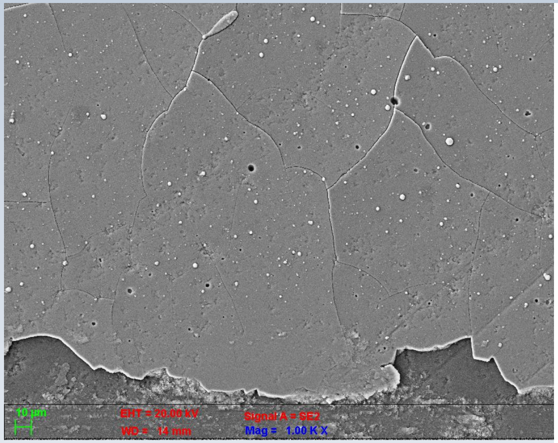
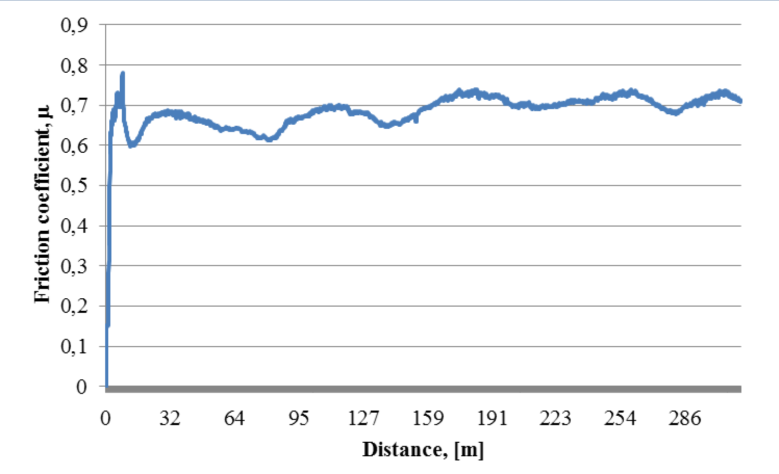
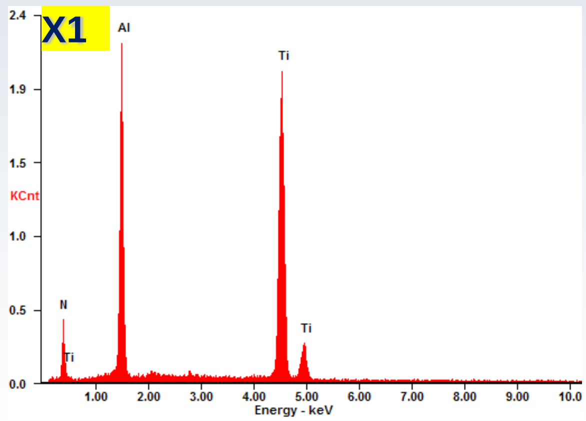
Coatings wear resistance









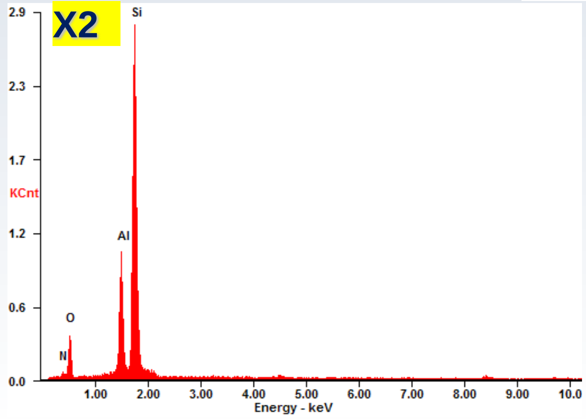
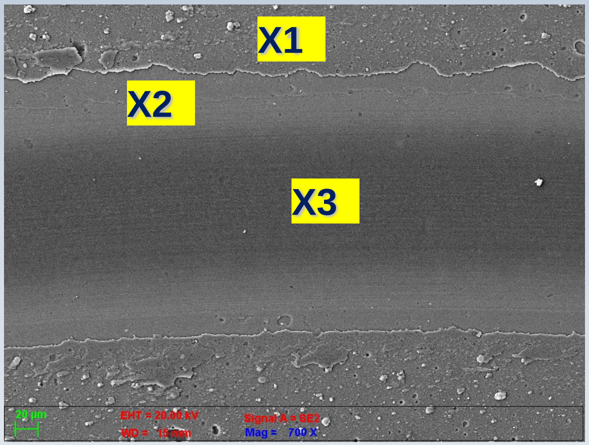
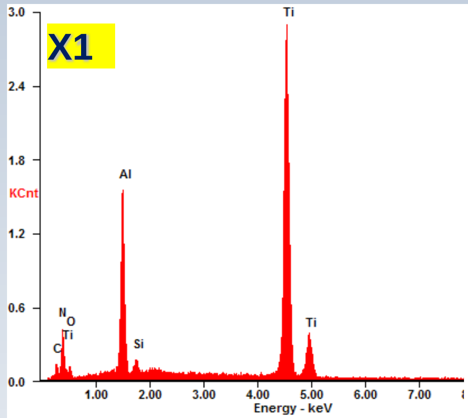


Diagram of friction coefficient according to the friction path during the pin-on-disc test for (Ti,Al)N coating deposited onto a sialon ceramics substrate

Trace of tribological damage on the surface of the (Ti,Al)N coating deposited onto a sialon ceramics substrate and diagrams of energy of backscatter X-ray radiation from the microarea: c) X1, d) X2

Coatings wear resistance





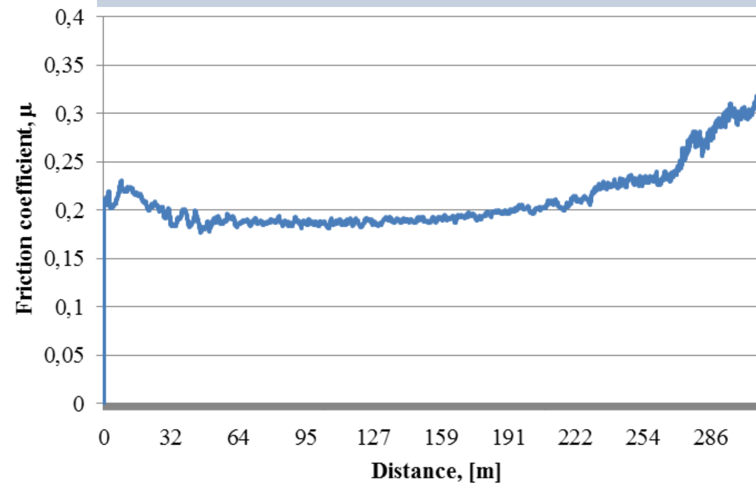
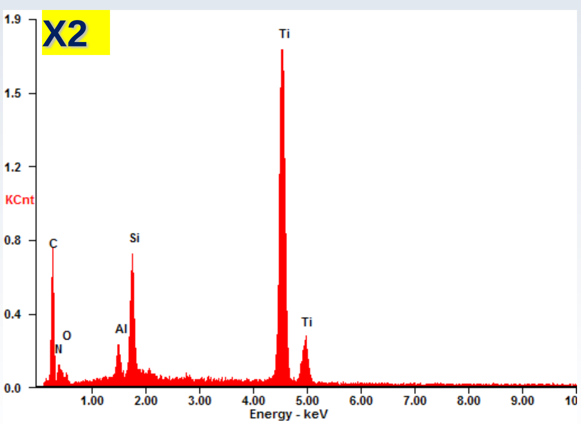
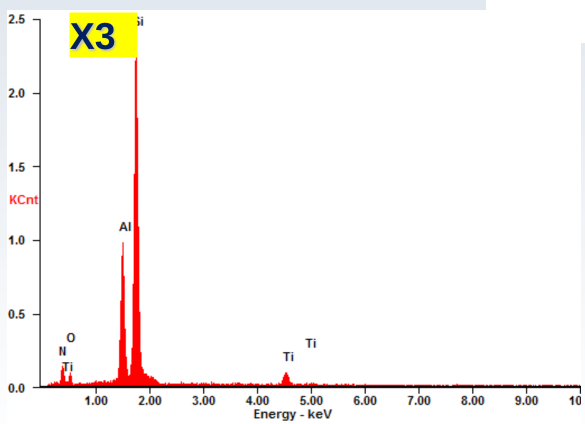


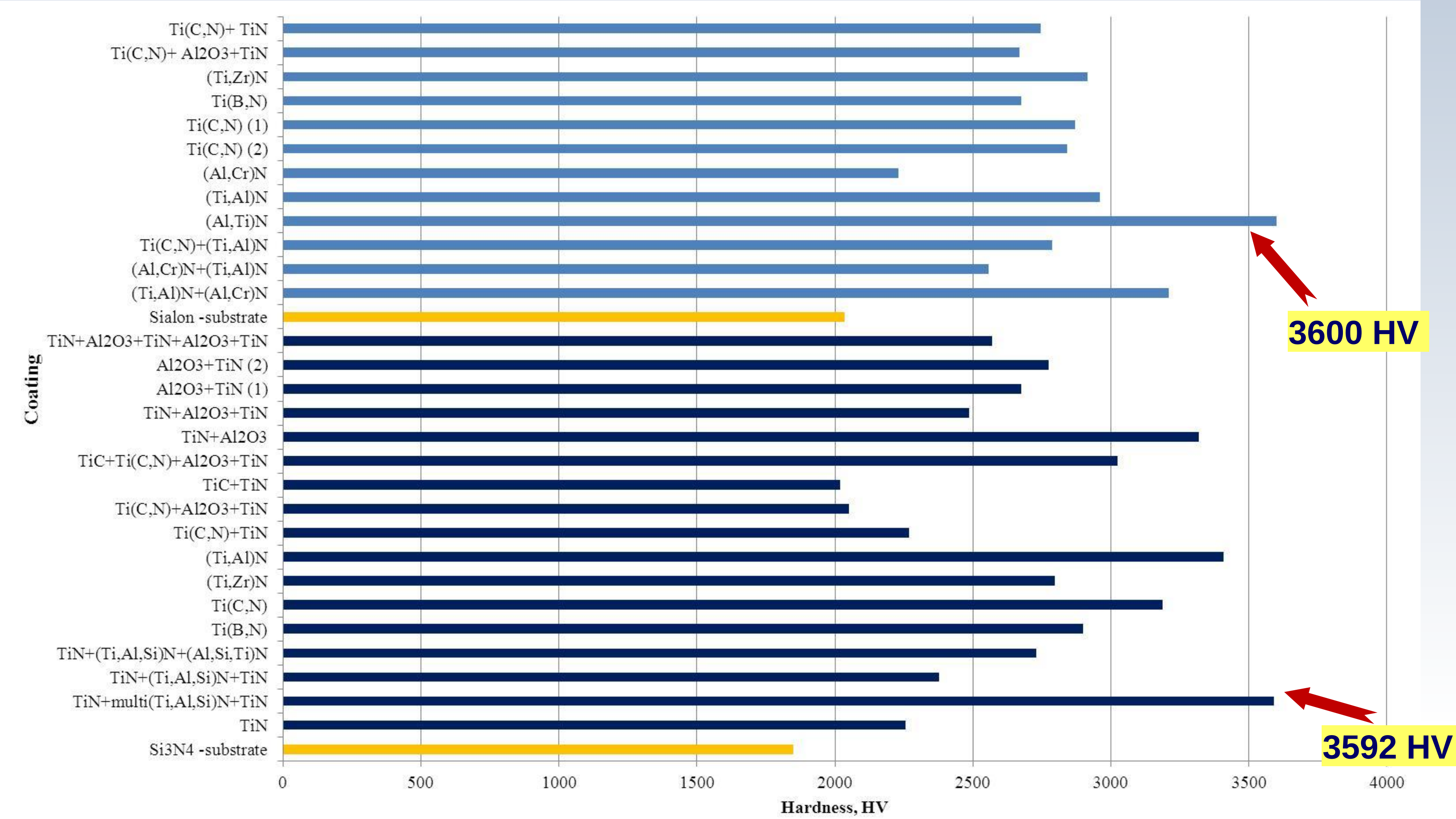
Diagram of friction coefficient according to the friction path during the pin-on-disc test for Ti(C,N)+(Ti,Al)N coating deposited onto a sialon ceramics substrate



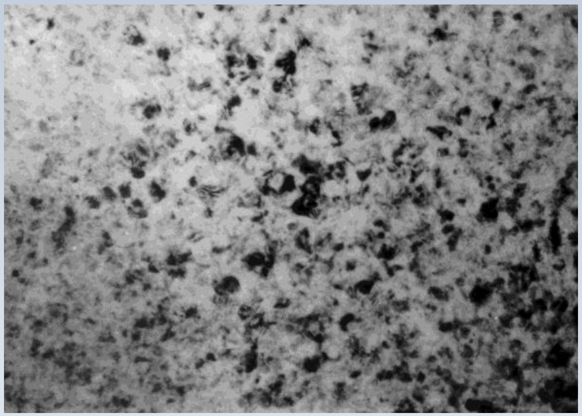


Trace of tribological damage on the surface of the Ti(C,N)+(Ti,Al)N coating deposited onto a sialon ceramics substrate and diagrams of energy of backscatter X-ray radiation from the microarea: b) X1, c) X2, d) X3

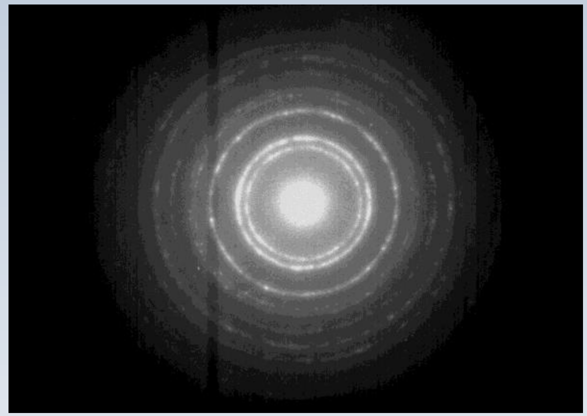
Coatings hardness



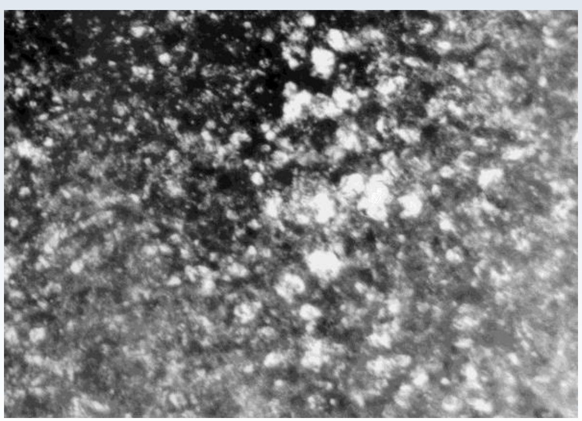
Structure of the TiN+multiTiAlSiN+TiN coating



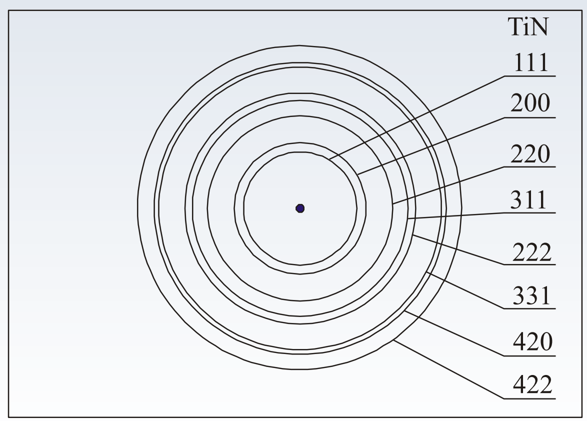
50 nm



Cross-section parallel to a layer surface

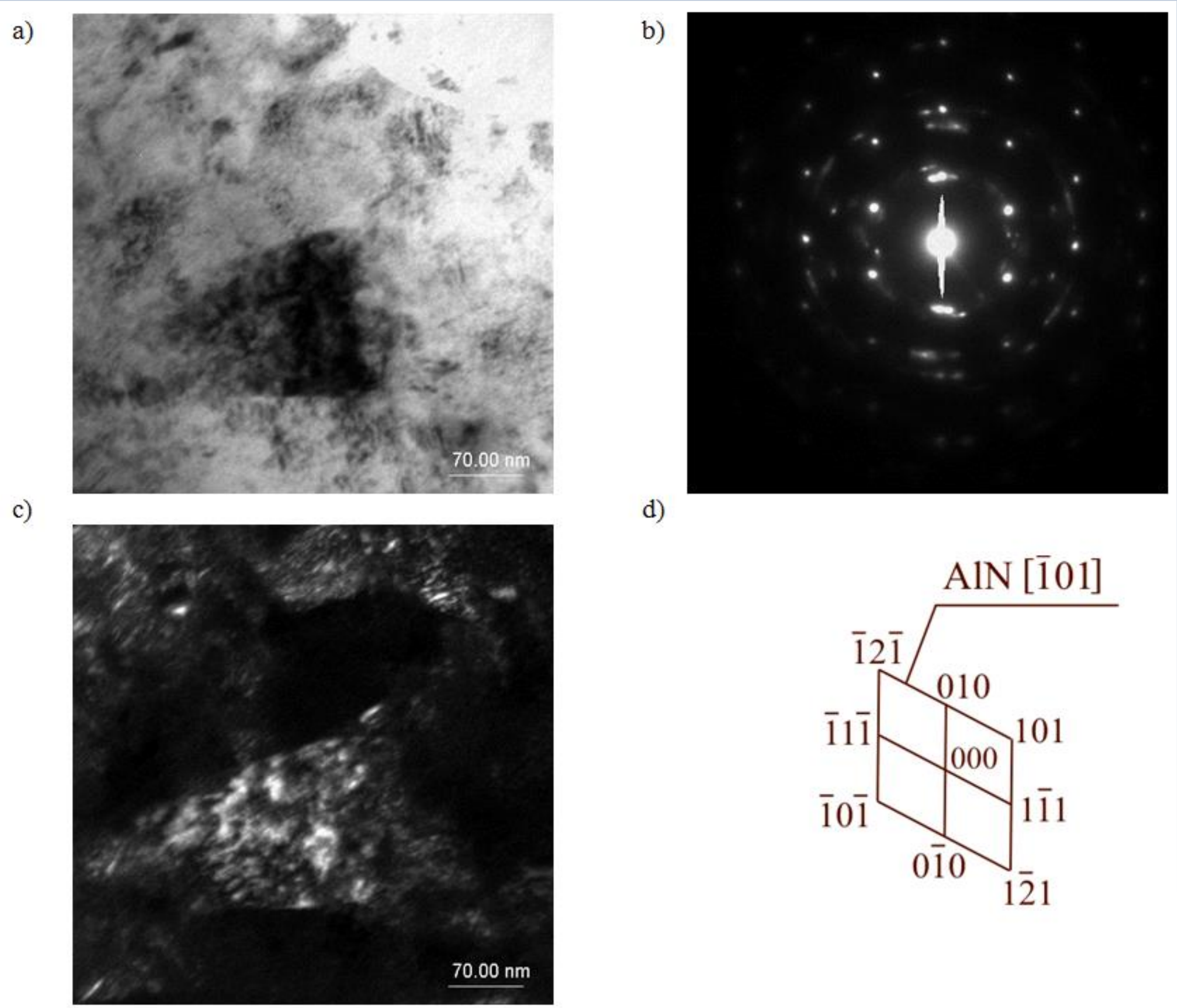


50 nm



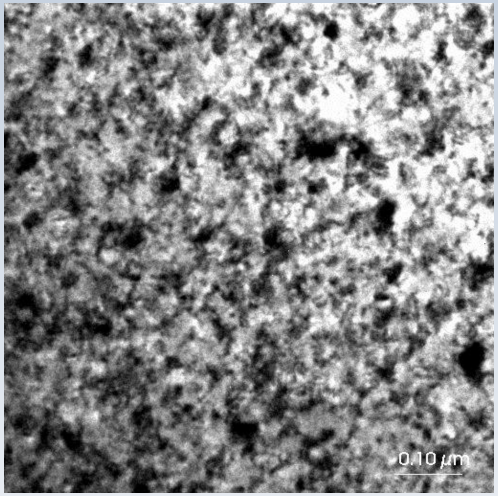
Dark field of reflexes (111) TiN

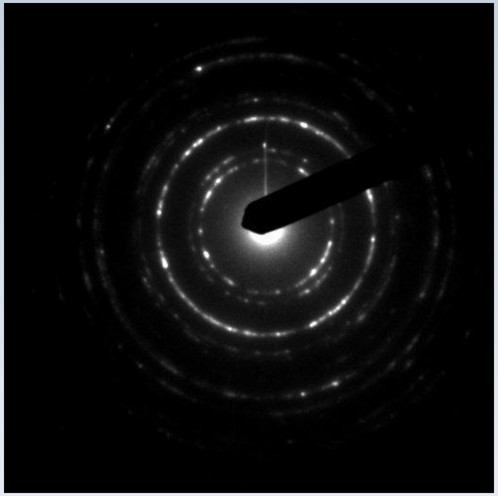
Structure of the (Al,Ti)N coating



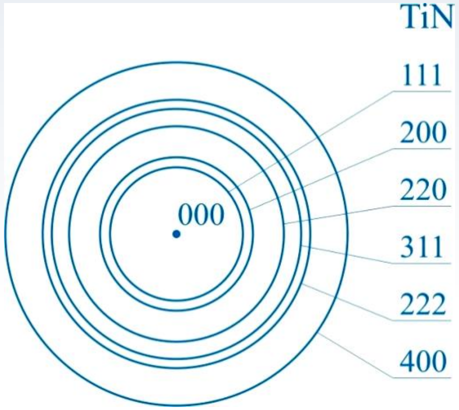
Cross-section parallel to a layer surface

Structure of the Ti(B,N) coating





Cross-section parallel to a layer surface



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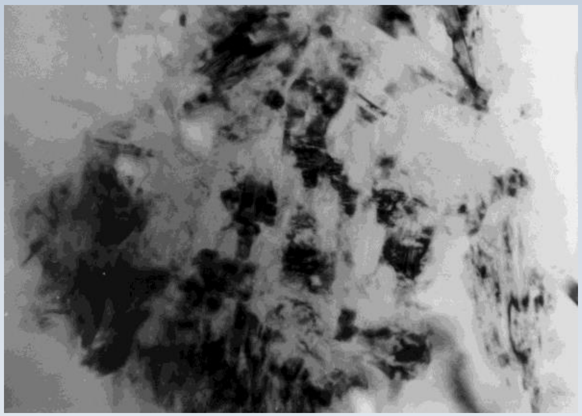
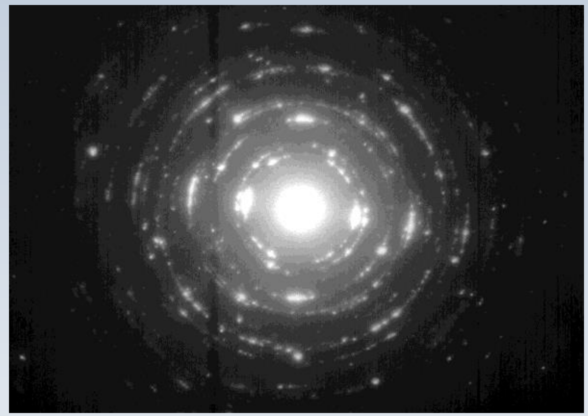
Structure of the $TiN+Al_2O_3$ coating

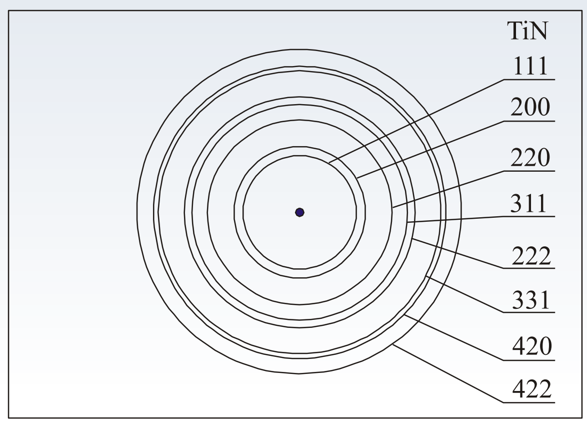


50 nm



Cross-section parallel to a layer surface

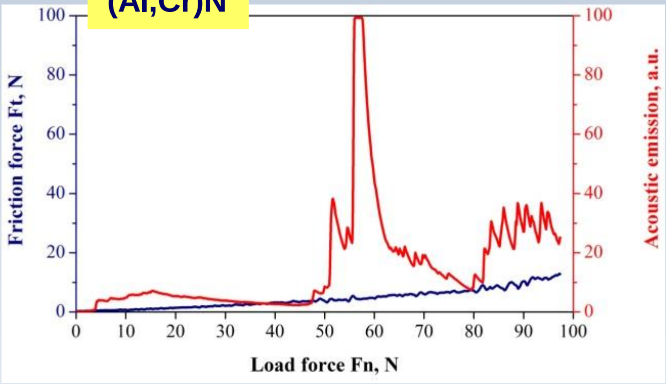
50 nm



Dark field of reflexes (111) TiN

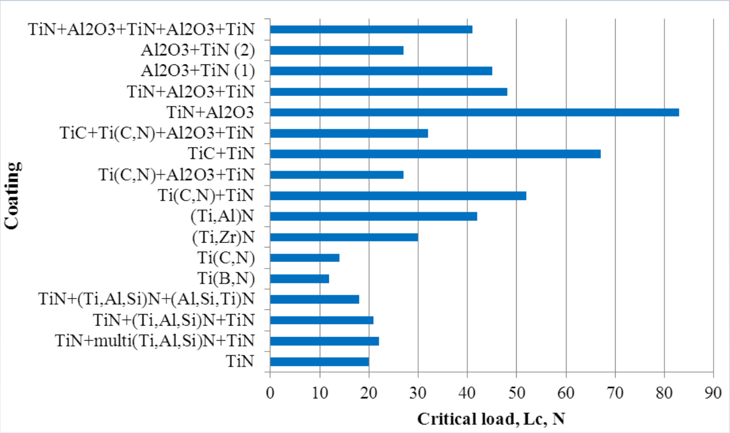
Coatings adhesion

(Al,Cr)N

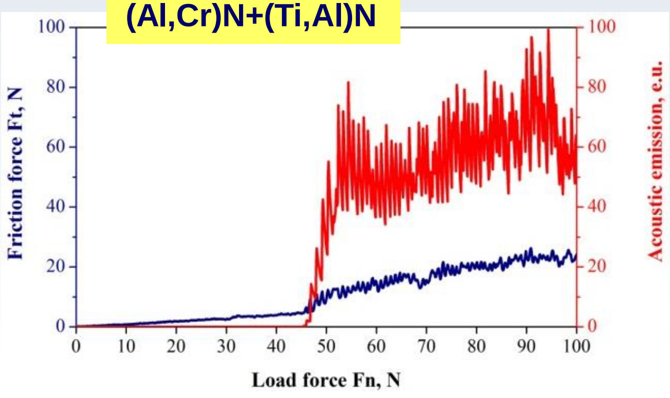


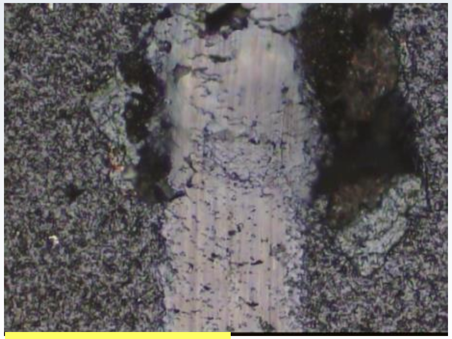
$L_{c\ (optical)}$= 53 N

Values of the critical load L_c for coatings deposited on nitride ceramics



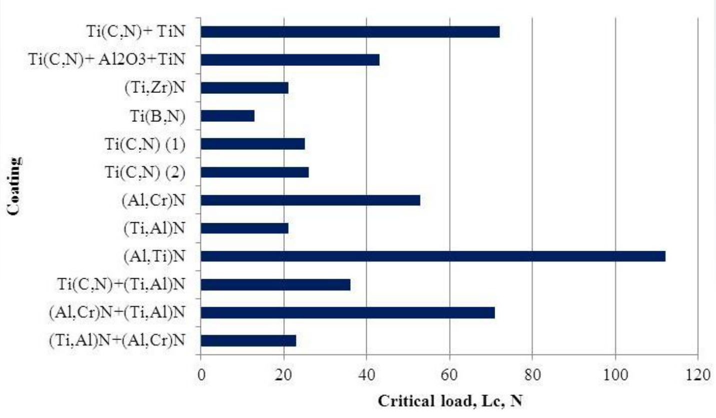
(Al,Cr)N+(Ti,Al)N



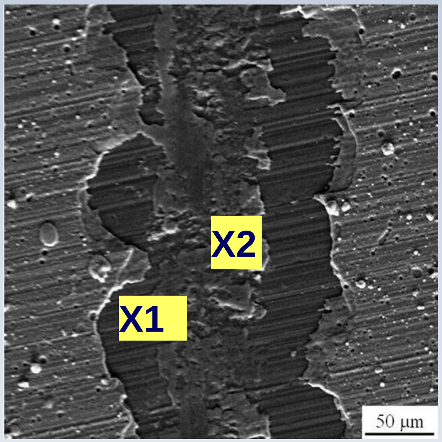


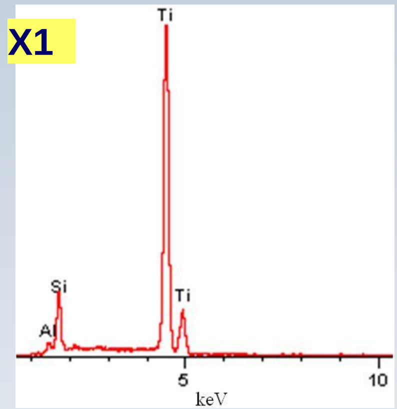
$L_{c\ (optical)}$ = 71 N

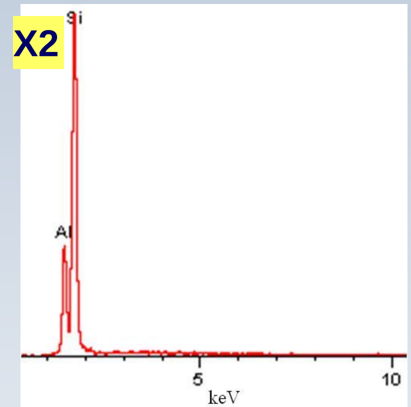
Values of the critical load L_c for coatings deposited on sialon tool ceramics



Coatings adhesion



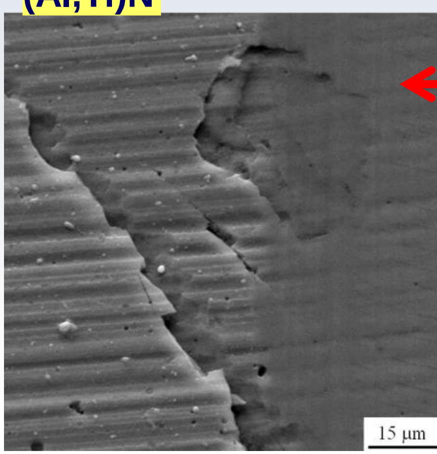




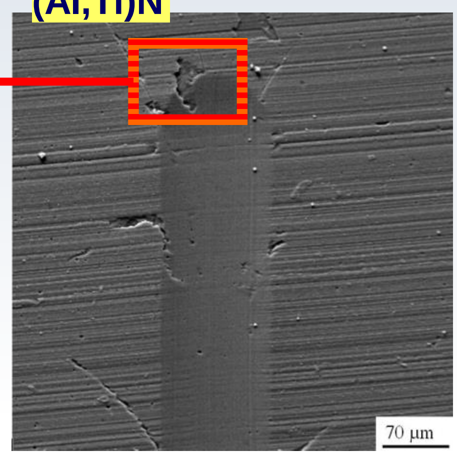
Indenter mark for over-critical load of the Ti(C,N) coating applied onto sialon tool ceramics.

Diagram of energy of backscatter X-ray radiation from the microarea: b) X1, c) X2

(Al,Ti)N



(Al,Ti)N



$Ti(C,N)+Al_2O_3+TiN$

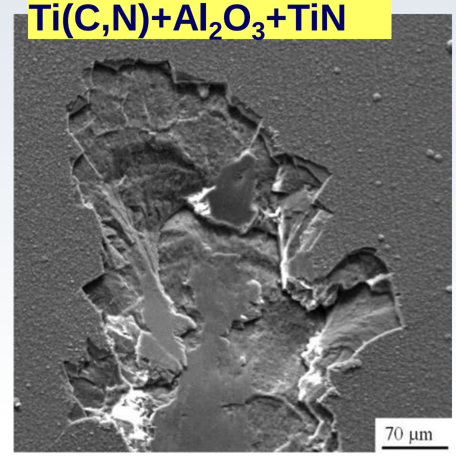
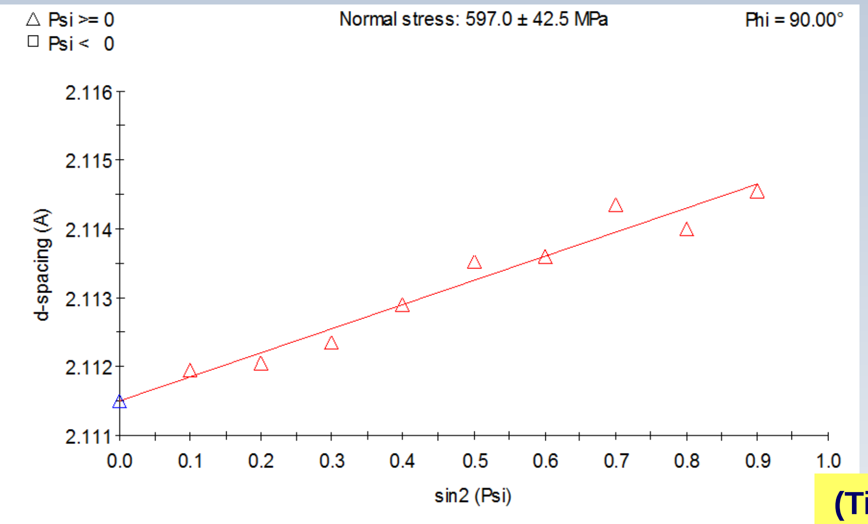
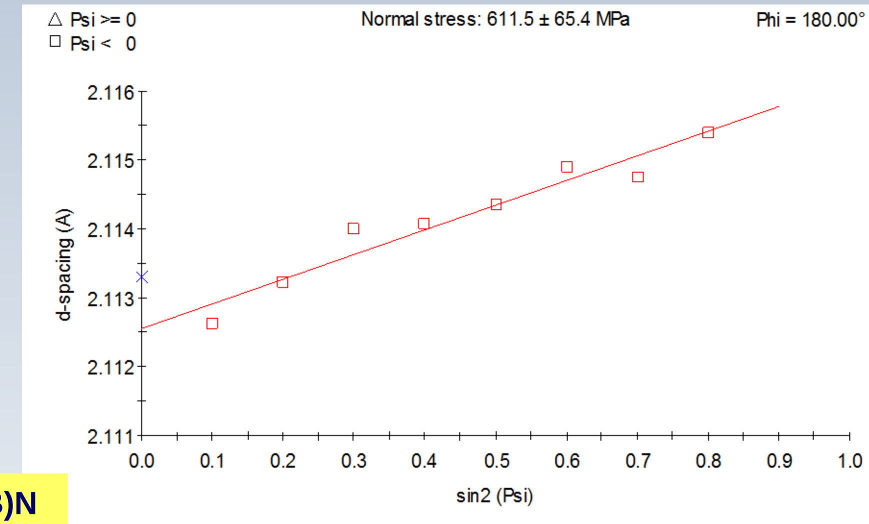


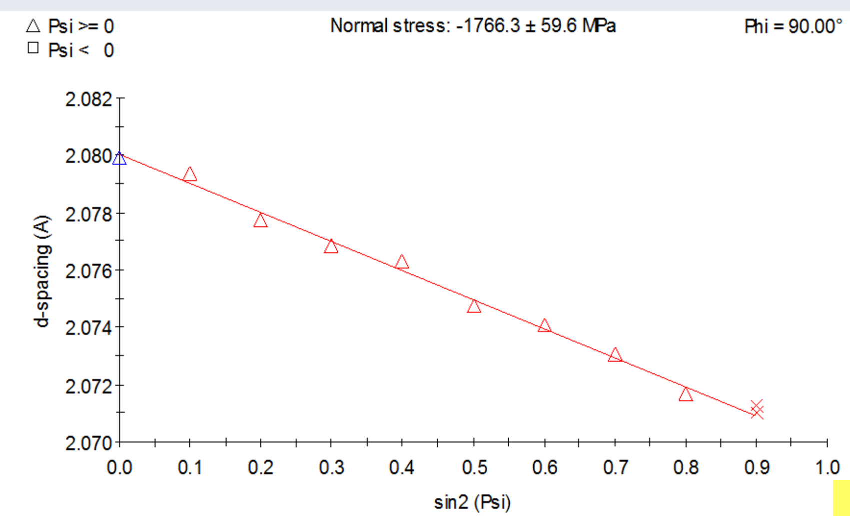
Image of damages caused as a result of a scratch test for coating deposited onto sialon tool ceramics

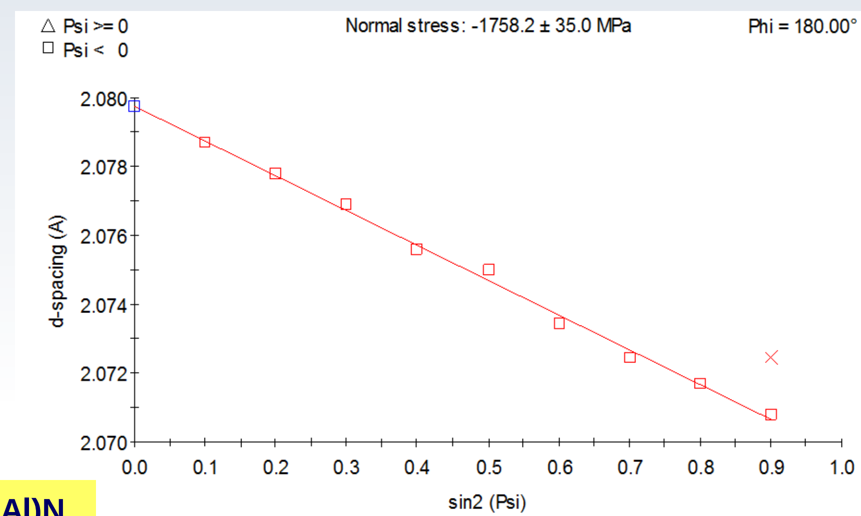
Coatings stresses





(Ti,B)N





(Ti,Al)N

Coatings stresses

Coatings	Substrate	Stresses, MPa	Standard deviation of stress, MPa
Ti(B,N)	Sialon ceramics	597.0 611.5	42.5 65.4
Ti(C,N) (1)	Sialon ceramics	-1102.4 -1212.9	39.8 42.8
(Ti,Al)N	Sialon ceramics	**-1766.3** **-1758.2**	**59.6** **35.0**
Ti(C,N)+Al_2O_3+TiN layer (Ti(C,N)	Sialon ceramics	616.6 610.4	37.5 32.3
Ti(C,N)+Al_2O_3+TiN layer Al_2O_3	Sialon ceramics	-594.5 -573.3	41.8 42.1
Ti(C,N)+TiN	Si_3N_4	379 302	49.2 38.3
Ti(C,N)+Al_2O_3+TiN	Si_3N_4	299 327	17.4 56.7
TiC+TiN	Si_3N_4	616 589	73.4 45.3
TiN+Al_2O_3	Si_3N_4	590 625	85.1 77.8
TiN+Al_2O_3+TiN*	Si_3N_4	1008 963	119.1 89.4
Al_2O_3+TiN (Iscar)*	Si_3N_4	915 879	43.3 54.9
TiN+Al_2O_3+TiN+Al_2O_3+TiN*	Si_3N_4	512 535	46.2 54.2

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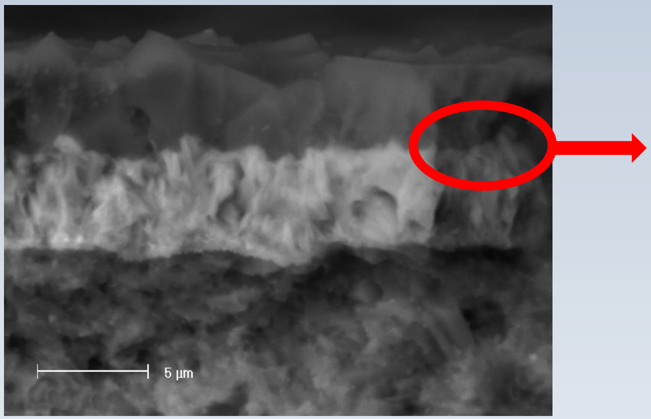
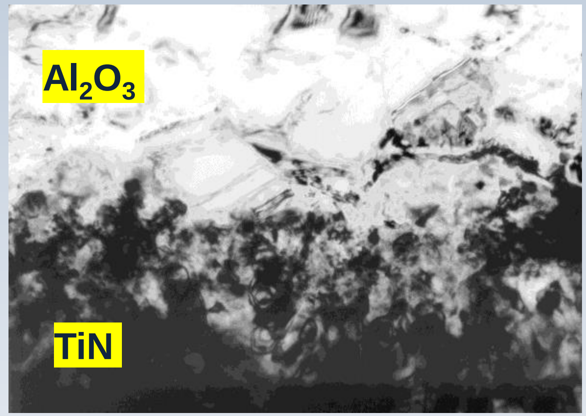
Structure of the TiN+Al_2O_3 coating

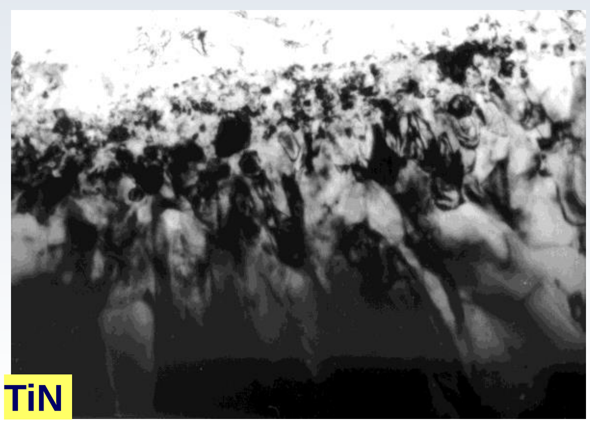




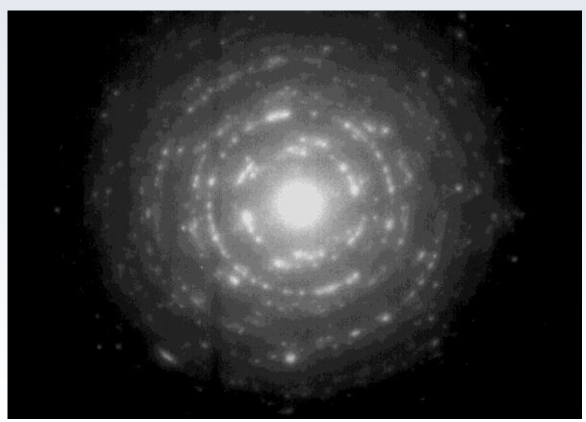
100 nm

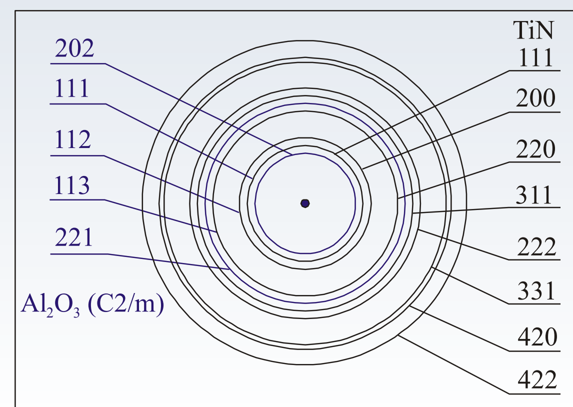
The transition zone

Cross-section perpendicular to a layer surface



200 nm





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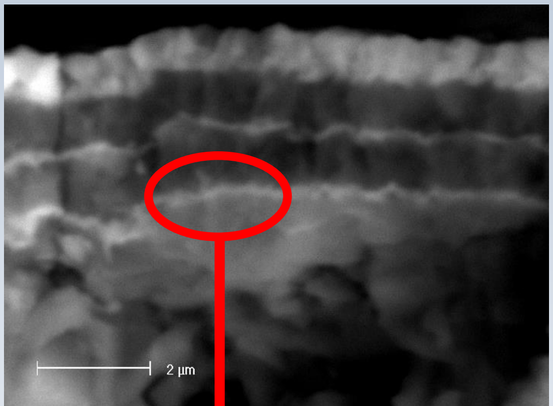
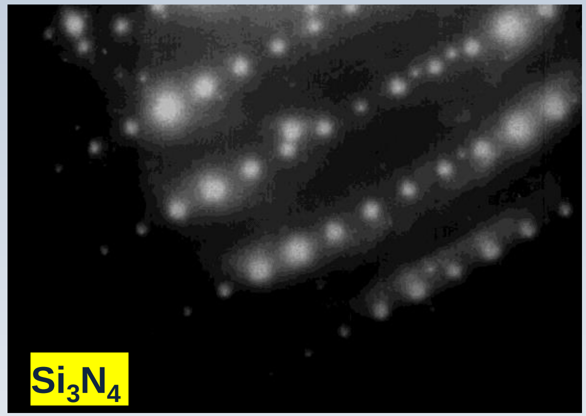
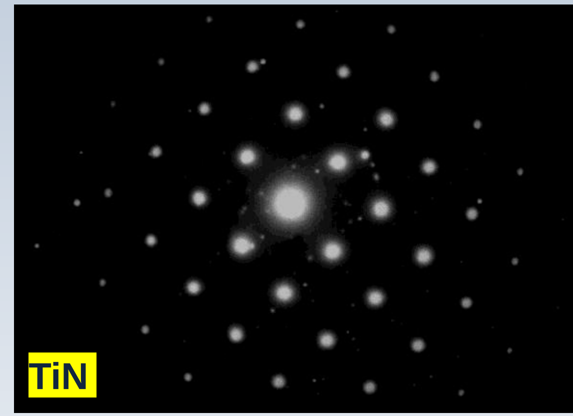
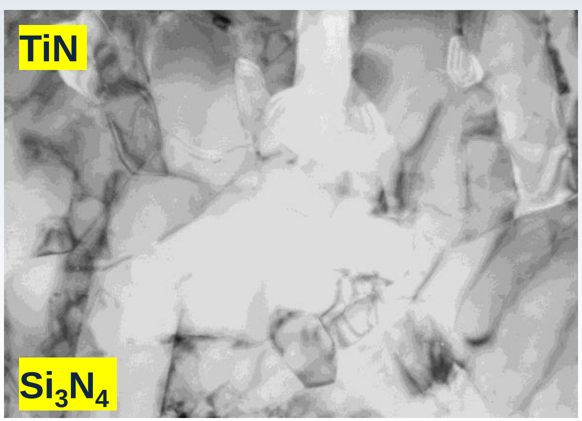
Structure of the $TiN+Al_2O_3+TiN+Al_2O_3+TiN$ coating



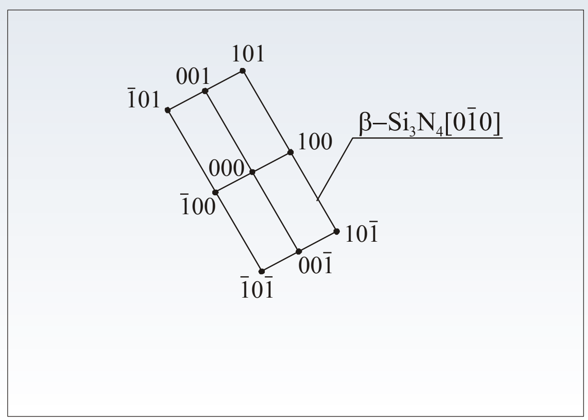


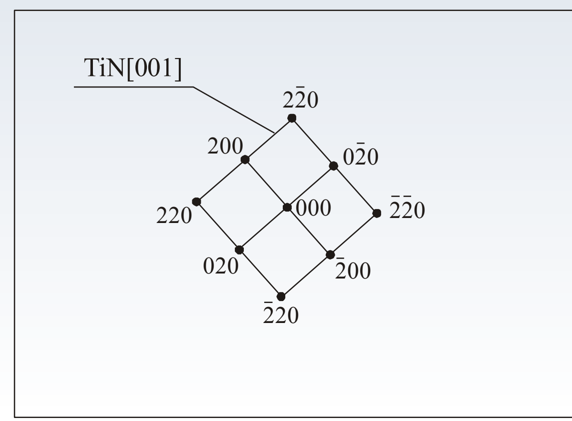


The transition zone



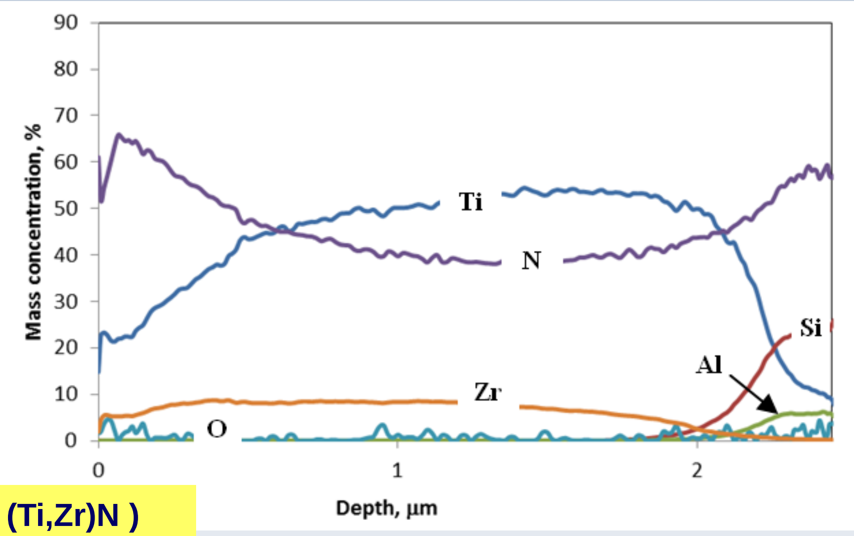
200 nm



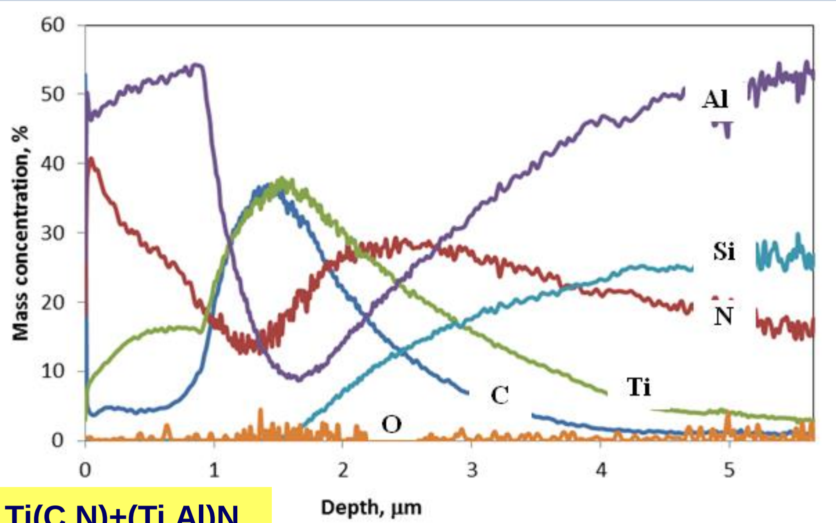


Cross-section perpendicular to a layer surface

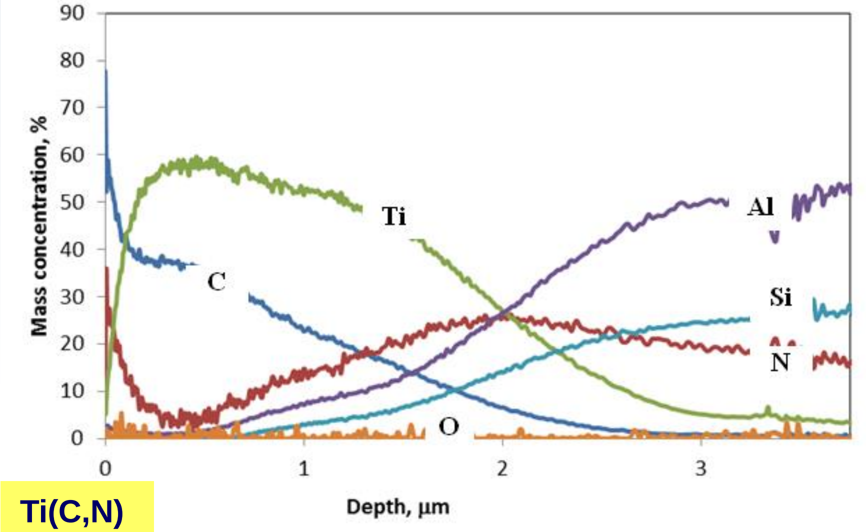
The chemical composition of coatings GDOES



(Ti,Zr)N)



Ti(C,N)+(Ti,Al)N

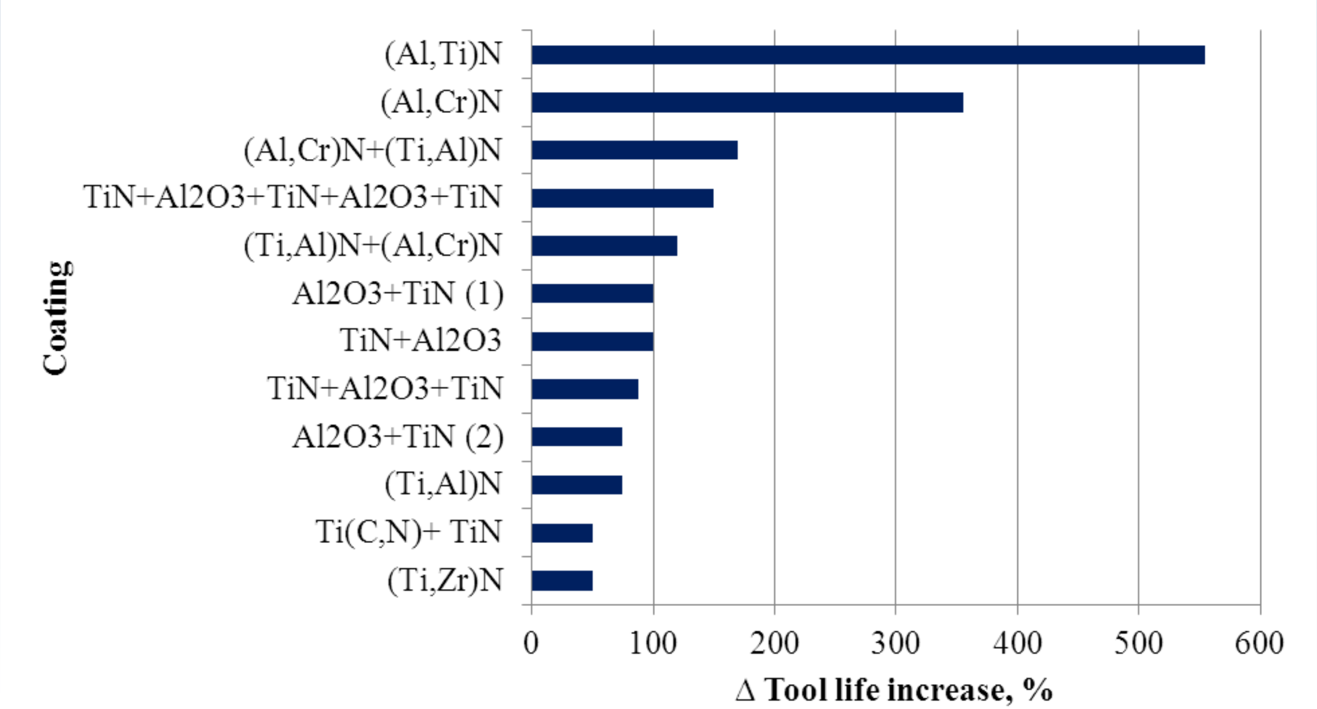


Ti(C,N)

It was found as a result of a GDOES analysis that a concentration of elements forming part of the substrate is increased in the bonding zone from the surface of coatings with a reduction, at the same time, in the concentration of elements forming part of the coating. This fact may be linked to the existence of a transition zone with of a diffusive character between the substrate material and the coating.

Increase in the life of ceramic cutting edges coated with PVD and CVD coatings versus uncoated tools

Investigations into the tool life allowed to select coatings with the best cutting properties:



exhibiting best cuttability, which is correlated with their high adhesion to the substrate and high hardness.

Conclusion

- The above-mentioned coatings improve the life of a cutting edge by up to 550% and for this reason, as well as due to a possibility of using them in environmental dry cutting processes without the use of process cooling and lubricating liquids, the coatings qualify for widespread industrial applications in cutting tools.

- Research methods used in this work (including cross-section TEM analysis) allow for a full characterization of the structure and properties of tool materials

- It is necessary for determining the influence of the technology and materials conditions on the increasing of their mechanical and functional properties.

Thank you
for your kind attention!