Tehnologie de realizare a acoperirilor multicomponent cu caracteristici functionale imbunatatite, prin utilizare a diverse structuri si compozitii (Partea a II-a)

Program NUCLEU
Proiect PN 19 06 01 03

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Obiectivul fazei

Realzarea de probe optimizate cu acoperiri tribologice uscate, cu structura de strat unic si compozitie constanta si evaluarea proprietatilor:

- **structurale si compozitionale** *(pe adancimea stratului depus)*

  - prin RBS - DFNA / IFIN-HH

- **compozitionale**

  - prin SEM-EDS - DFNA / IFIN-HH

- **mecanice si tribologice**

  - Aderenta la substrat - prin Scratch Test-UTBv - Fac. Stiinta Materialelor,
  - Duritatea - prin Hardness Test-UTBv - Fac. Stiinta Materialelor,
  - Caracteristicile tribologice - prin Ball on Disck Tribometer Test-UTBv
  - Fac. Stiinta Materialelor,

Acoperiri tribologice – Caracteristici esentiale si parametrii de evaluare

1. Coeficient de frecare/COF scăzut, determinat prin testul tribometric *(Pin/ball-on disk Tribometer Test),* in micro-tribologie este o masura a fortei de frecare *(respectiv a fortelor de abraziune si de adeziune in nano-tribologie)* ce se opune miscării unui obiect aflat in contact cu o suprafată *(previzionat de **COF** al materialelor utilizate si de structuri super-nanostructurate)*

2. Duritate ridicată, definita ca rezistenta la deformare *(prin zgariere sau indentare/ amprentare)* sub actiunea unui corp strain, este determinata uzual prin testul de indentare *(Hardness Indentation Test)* si se masoara in **GPa**. Pentru indentare: $H = P/\alpha_0.a^2$, unde: P = forta de amprentare; a = dimensiunea amprentei lasate de indentor; α_0 = constanta *(previzionat de **H** al materialelot utilizate si de strcturi super-nanostructurate)*

3. Tenacitate *(toughness = K_c)* ridicată sau rezistenta la fracturare, este data de relatia: $K_c = \alpha_1(E/H)^{1/2}(P/c^{3/2})$, unde: P=sarcina de deformare; c= dimensiunea fisurii rezultate prin indentare; α_1=coeficient numeric care tine cont de dimensiunile indentorului: *α_1=0,016 pentru varfurile de indentare, tip Berkovich si Vikers (este previzionata de parametrii **σ_r, E** si **H** ai materialelor utilizate si de structure super-nanostructurate)*

4. Aderență bună la substrat si coeziune ridicata intre straturi, este determinata uzual prin testul de zgariere *(Scratch Test)* si este redata de sarcinile critice **L_{C1}, L_{C2} si L_{C3},** ce reprezinta fortele (*in Newton)* la care apar la: Prima fisură **(L_{C1});** Prima delaminare **(L_{C2});** Delaminare totala ***(**Exfoliere a mai mult de 50% din strat -**L_{C3},** previzionata de Electronegativitatea materialelor utilizate- χ si de parametrii procesului tehnologic)*

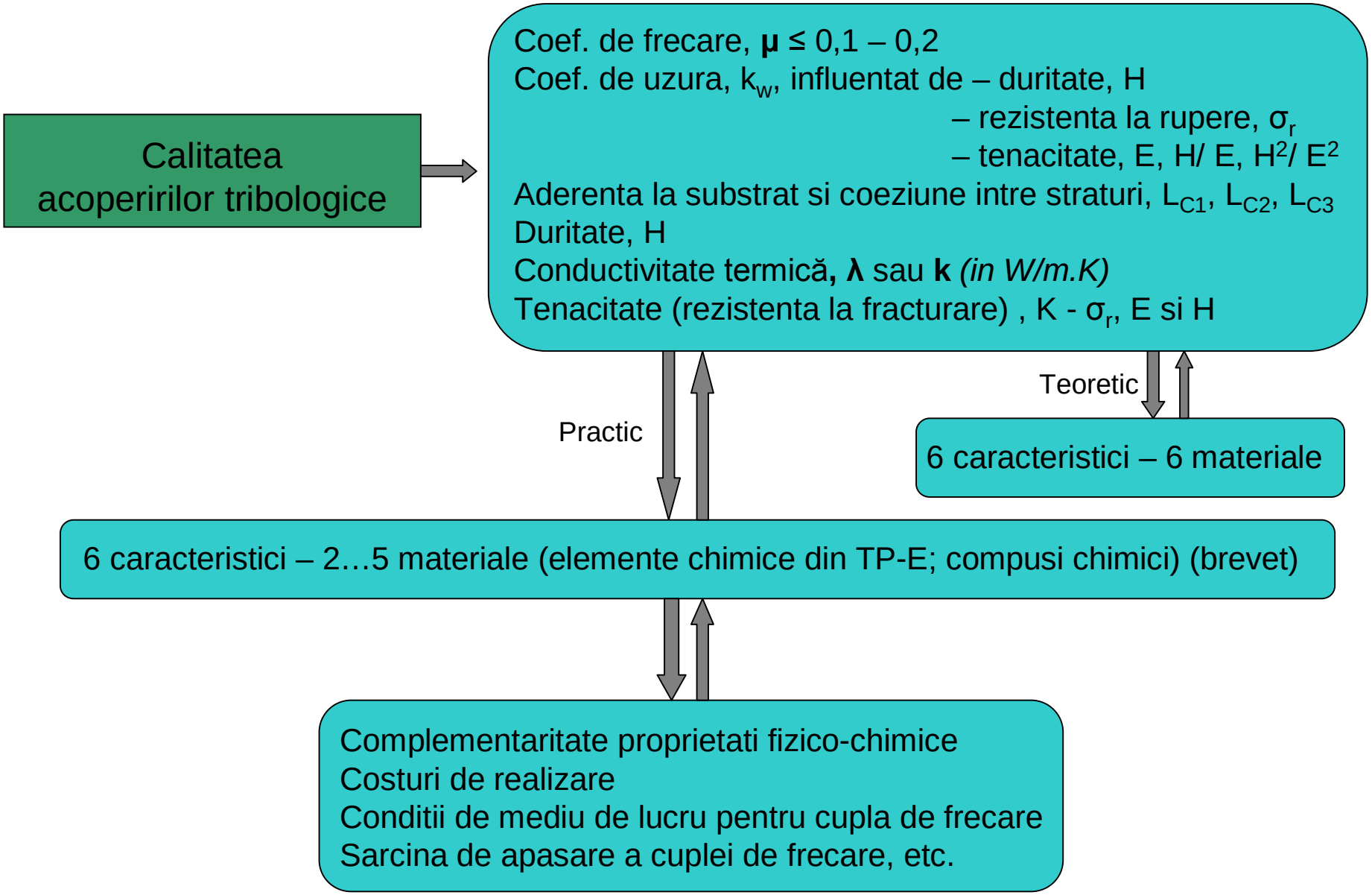
5. Conductivitate termică *ridicată,* notata prin **λ** sau **k** *(in W/m.K) (previzionata de coeficientul **λ** al materialelor utilizate si de combinarea lor sinergica, in cazul acoperirilor super-nanostructurate);*

6. Rezistență înaltă la coroziune şi oxidare termică/ stabilitate termică şi chimică, *(previzionata de parametrul χ al materialelor utilizate si de combinarea lor sinergica, in super-nano-structuri).*

*NOTA: Combinarea sinergica a proprietatilor materialelor utilizate, conform CBI 00167/2016 are loc in cazul acoperirilor cu structuri super-nanostructurate, adica in **super-nano-structuri cu grosimi mai mici de 10 nm***

CoF static/ dinamic/ cu lubrifiere - pentru materiale comune

Materials		Static Friction,		Kinetic/Sliding Friction,	
		Dry and clean	Lubricated	Dry and clean	Lubricated
Aluminium	Steel	0.61		0.47	
Aluminium	Aluminium	1.05–1.35	0.3	1.4–1.5	
Gold	Gold			2.5	
Platinum	Platinum	1.2	0.25	3.0	
Silver	Silver	1.4	0.55	1.5	
Alumina ceramic	Silicon nitride ceramic				0.004 (wet)
BAM (Ceramic alloy $AlMgB_{14}$)	Titanium diboride (TiB_2)	0.04–0.05	0.02		
Brass	Steel	0.35–0.51	0.1	0.44[1]	
Cast iron	Copper	1.05		0.29	
Cast iron	Zinc	0.85		0.21	
Concrete	Rubber	1.0	0.30 (wet)	0.6–0.85	0.45–0.75 (wet)
Concrete	Wood	0.62			
Copper	Glass	0.68		0.53	
Copper	Steel	0.53		0.36	0.18
Glass	Glass	0.9–1.0	0.005–0.01	0.4	0.09–0.116
Human synovial fluid	Human cartilage		0.01		0.003
Ice	Ice	0.02–0.09			
Polyethene	Steel	0.2	0.2		
PTFE (Teflon)	PTFE (Teflon)	0.04	0.04		0.04
Steel	Ice	0.03			
Steel	PTFE (Teflon)	0.04–0.2	0.04		0.04
Steel	Steel	0.74–0.80	0.005–0.23	0.42–0.62	0.029–0.19
Wood	Metal	0.2–0.6	0.2 (wet)	0.49	0.075
Wood	Wood	0.25–0.62	0.2 (wet)	0.32–0.48	0.067–0.167


Calitatea acoperirilor tribologice
Coef. de frecare, μ ≤ 0,1 – 0,2
Coef. de uzura, k_w, influentat de – duritate, H
– rezistenta la rupere, σ_r
– tenacitate, E, H/ E, H^2/ E^2
Aderenta la substrat si coeziune intre straturi, L_{C1}, L_{C2}, L_{C3}
Duritate, H
Conductivitate termică, λ sau k (in W/m.K)
Tenacitate (rezistenta la fracturare) , K - σ_r, E si H
Practic
Teoretic
6 caracteristici – 6 materiale
6 caracteristici – 2…5 materiale (elemente chimice din TP-E; compusi chimici) (brevet)
Complementaritate proprietati fizico-chimice
Costuri de realizare
Conditii de mediu de lucru pentru cupla de frecare
Sarcina de apasare a cuplei de frecare, etc.

Materiale utilizate in cadrul fazei

Tab. 1**. Proprietațile **Ti, TiB_2, WC, WS_2, TiN, B_4C

Materialul tintelor de pulverizare	**E** [GPa]	**H** [GPa]	**λ** [W/m.K]	**μ/COF** *(fata de otel)*
Grupa Principala - 1				
WC	450 – 650	2,1	80-100	0,4-0,6
TiB_2	510 - 550	33	25	0,5 – 0,65
WS_2	400 - 410	1	32 - 140	0,03-0,07
Ti	110	0,83 - 3,42	22	0,4-0,6
Grupa Principala - 2				
B_4C	218-290	41-45	687	0,35 – 0,55
TiB_2	510 - 550	33	25	0,5 – 0,65
WS_2	400 - 410	1	32-140	0,03-0,07
TiN	250 - 320	31	19; 29	0,18-0,51

Date ale structurii nou create, tip HEM_5		
Elementul chimic	Strucura cristalina	Raza atomica [pm]
W	Cub cu fete centrate/ CFC	193
Ti	hexagonal compact/ hcp	176
C	Hexagonal	67
B	Romboidal	87
S	Ortorombic	88
Date ale structurii nou create, tip HEM_6		
W	CFC	193
Ti	hcp	176
C	Hexagonal	67
B	Romboidal	87
S	Ortorombic	88
N	-	56

Tetraboron carbide (B_4C)

Boron carbide (chemical formula approximately B_4C) is an extremely hard boron–carbon ceramic and covalent material used in tank armor, bulletproof vests, engine sabotage powders,[1] as well as numerous industrial applications. With a Vickers hardness of >30 GPa, it is one of the hardest known materials, behind cubic boron nitride and diamond.[2]00

Vickers hardness of selected hard materials	
Material	Vickers hardness (GPa)
Diamond	115
c-BC_2N	76
γ-Boron	58
c-BN	48
OsB_4	37
B_4C	35
WB_4	~30
$AlMgB_{14}$	26.7
ReB_2	~20

Titanium diboride (TiB_2)

Titanium diboride (TiB_2) is an extremely hard ceramic which has excellent heat conductivity, oxidation stability and wear resistance. TiB_2 is also a reasonable electrical conductor

Physical properties
TiB2 shares some properties with Boron Carbide & Titanium carbide, but many of its properties are superior to those of B4C & TiC

Exceptional hardness at extreme temperature
2nd hardest material at 3000°C (# Diamond)
3rd hardest material at 2800°C (# cBN)
4th hardest material at 2100°C (# B4C)
5th hardest material at 1000°C (# B6O)

Advantages over other borides
Highest Boride Elastic Modulus
Highest Boride Fracture Toughness
Highest Boride Compressive strength
2nd highest Boride melting point (3225 °C) (# HfB2)

Other advantages
High thermal conductivity (60-120 W/(m K)),
High electrical conductivity (~105 S/cm)

Titanium nitride (TiN)

Titanium nitride (TiN; sometimes known as Tinite) is an extremely hard ceramic material, often used as a coating on titanium alloys, steel, carbide, and aluminium components to improve the substrate's surface properties.

Applied as a thin coating, TiN is used to harden and protect cutting and sliding surfaces, for decorative purposes (due to its golden appearance), and as a non-toxic exterior for medical implants. In most applications a coating of less than 5 micrometres (0.00020 in) is applied.

TiN has a Vickers hardness of 1800–2100, a modulus of elasticity of 251 GPa, a thermal expansion coefficient of 9.35×10^{-6} K^{-1}, and a superconducting transition temperature of 5.6 K.

TiN will oxidize at 800 °C in a normal atmosphere. TiN has a brown color, and appears gold when applied as a coating. It is chemically stable at 20 °C, according to laboratory tests, but can be slowly attacked by concentrated acid solutions with rising temperatures.

Depending on the substrate material and surface finish, TiN will have a coefficient of friction ranging from 0.4 to 0.9 against another TiN surface (non-lubricated).

The typical TiN formation has a crystal structure of NaCl-type with a roughly 1:1 stoichiometry; TiN_x compounds with *x* ranging from 0.6 to 1.2 are, however, thermodynamically stable.

Tungsten disulfide (WS_2) - Lower Friction Lubricant

https://www.lowerfriction.com/products/powder

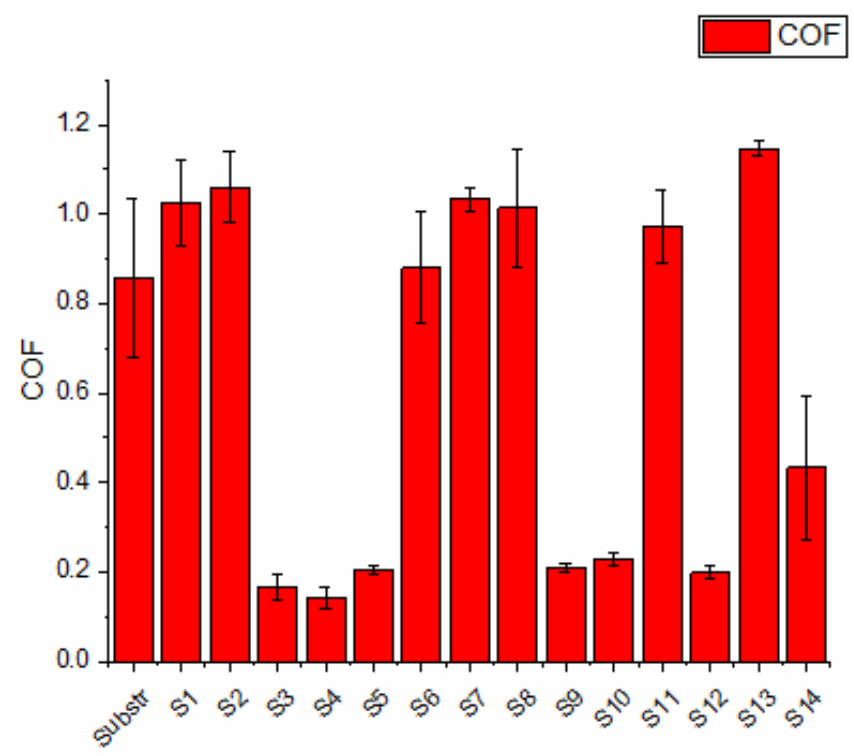
•Tungsten Disulfide (WS2) is dry/solid lubricant powder and is one of the most lubricious substance in world. WS2 offers excellant dry lubricity (COF: 0.03) unmatched to any other substance, including Graphite or Molybdenum Disulfide (MoS2).

•Tungsten Disulfide (WS2) can also be used in high temperature and high pressure applications. It offers temperature resistance from -450 deg F (-270º C) to 1200 deg F (650º C) in normal atmosphere and from -305 deg F (-188º C) to 2400º F (1316º C) in Vacuum. Load bearing ability of coated film is extremely high at 300,000 psi.

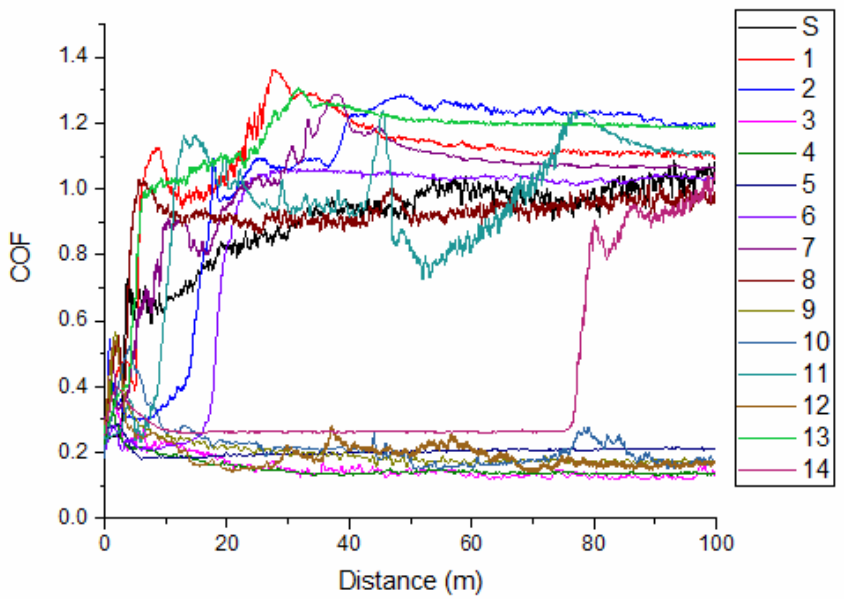
•Tungsten Disulfide (WS2) can also be used instaed of Molybdenum Disulfide (MoS2). See comparison of WS2 / MoS2

•Since the powder offers one of the lowest Coefficient of Friction (Dynamic @ 0.03 & Static @ 0.07), the applications are unlimited and could be tried with every conceivable idea.

Rezultatele testului de tribologie/CoF – pentru probe realizate in Faza 1



a) Coeficientul mediu de frecare



b) Variatia COF cu distanta de alunecare

Probe cu CoF mai mic de 0,3 - pentru optimizare

Proba 3: **WC**-120W + **TiB2**-270W + **Ti**-120W + **WS2**-100W

Proba 4: **WC**-180W + **TiB2**-180W + **Ti**-60W + **WS2**-100W

Proba 5: **TiB2**-30W + **Ti**-30W + **WS2**-110W

Proba 9: **B4C**-60W + **TiB2**-90W + **TiN**-60W + **WS2**-100W

Proba 10: **B4C**-xW + **TiB_2**-yW + **TiN**-zW + **WS_2**-100W

Proba 12: **B4C**-36W + **TiB2**-180W + **TiN**-36W + **WS2**-100W

Parametrii Tehnologici de realizare:

1.Probe optimizate, realizate in Faza 1 a proiectului: 9; 10; 12.

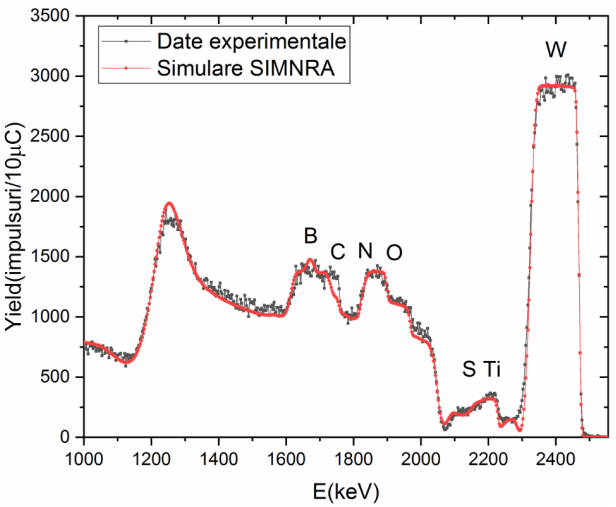
2. Probe noi, realizate in cadrul Fazei 2 a proiectului

Tip de proba	Nr. Proba	Tipul de strat, Structura si Compozitia stratului depus (Materialele depuse si puterile absorbite in procesul de pulverizare)	Timp depunere [h]
Selectate pentru optimizare	9	**Strat unic cu compozitie constanta** **B4C/G1**-60W + **TiB2/G2**-90W + **TiN/G4**-60W + **WS2/G3**-100W	3,00
	10	**Multistrat din 8 pachete repetitive cu compozitie modulata in pachet** [(**B4C**-24W + **TiB**2-240W + **TiN**-24W + **WS2**-100W) -5 minute + (**B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-100W) - 5 minute + (**B4C**-348W + **TiB**2-120W + **TiN**-72W + **WS2**-100W) – 5 minute]	3,00
	12	**Strat unic cu compozitie constanta** **B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-100W	2,00
Optimizate	15	**Strat unic cu compozitie constanta** **B4C**-36W + **TiB**2-60W + **TiN**-36W + **WS2**-60W	3,00
	16	**Strat unic cu compozitie constanta** **B4C**-36W + **TiB**2-60W + **TiN**-36W + **WS2**-80W	3,00
	17	**Strat unic cu compozitie constanta** **B4C**-36W + **TiB**2-60W + **TiN**-36W + **WS2**-120W	3,00
	18	**Strat unic cu compozitie constanta** **B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-120W	3,00
	19	**Multistrat din 12 pachete identice si repetitive, cu compozitie modulata in pachet** **Pachet:** [(**B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-120W)/-5 minute + (**B4C**-36W + **TiB**2-120W + **TiN**-36W + **WS2**-80W) - 5 minute + (**B4C**-36W + **TiB**2-60W + **TiN**-36W + **WS2**-40W) – 5 minute]	3,00
	20	**Multistrat din 12 pachete identice si repetitive, cu compozitie modulata in pachet** **Pachet:** [(**B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-40W)/-5minute + (**B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-80W)-5minute + (**B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-120W)]-5minute]	3,00

Compozitia probelor realizate in Faza 1 si 2 a proiectului (9, 10, 12 si 15 – 19), obtinuta prin RBS

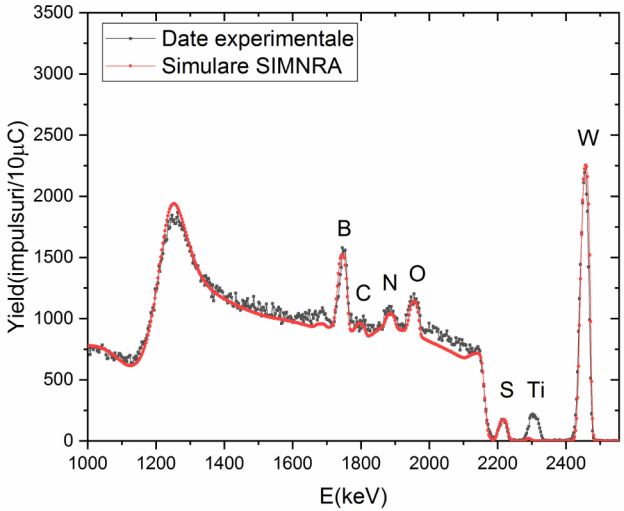
Proba Nr.	Formule Stoechiometrice in adancimea acoperirii	Grosimea – in TFU
9	$W_{0.185}Ti_{0.1}B_{0.231}S_{0.225}C_{0.02}O_{0.15}N_{0.089}$	10000
	$W_{0.17}Ti_{0.1}B_{0.234}S_{0.22}C_{0.02}O_{0.135}N_{0.121}$	4255
10	$W_{0.16}Ti_{0.11}B_{0.231}S_{0.25}C_{0.02}O_{0.14}N_{0.089}$	10000
	$W_{0.14}Ti_{0.11}B_{0.234}S_{0.25}C_{0.02}O_{0.125}N_{0.121}$	2600
	$W_{0.08}Ti_{0.11}B_{0.252}S_{0.19}C_{0.02}O_{0.277}N_{0.071}$	333
	$W_{0.17}Ti_{0.11}B_{0.272}S_{0.07}C_{0.02}O_{0.217}N_{0.141}$	777
	$W_{0.1}Ti_{0.08}B_{0.254}S_{0.12}C_{0.02}O_{0.256}N_{0.17}$	3555
	$W_{0.074}Ti_{0.11}B_{0.272}S_{0.07}C_{0.02}O_{0.313}N_{0.141}$	3000
12	$W_{0.16}Ti_{0.11}B_{0.231}S_{0.25}C_{0.02}O_{0.14}N_{0.089}$	5780
	$W_{0.15}Ti_{0.11}B_{0.254}S_{0.25}C_{0.02}O_{0.095}N_{0.121}$	2755
	$W_{0.14}Ti_{0.11}B_{0.272}S_{0.19}C_{0.02}O_{0.197}N_{0.071}$	2111
15	$W_{0.16}Ti_{0.1}S_{0.17}Al_{0.01}O_{0.34}N_{0.03}C_{0.04}B_{0.15}$	4500
	$W_{0.128}Ti_{0.02}S_{0.15}Al_{0.01}O_{0.44}N_{0.03}C_{0.04}B_{0.182}$	4000
16	$W_{0.19}Ti_{0.04}S_{0.22}Al_{0.05}O_{0.25}N_{0.03}C_{0.06}B_{0.16}$	4000
	$W_{0.19}Ti_{0.04}S_{0.22}Al_{0.05}O_{0.25}N_{0.03}C_{0.04}B_{0.18}$	4000
	$W_{0.18}Ti_{0.04}S_{0.18}Al_{0.05}O_{0.32}N_{0.03}C_{0.04}B_{0.16}$	2000
17	$W_{0.2}Ti_{0.05}S_{0.22}Al_{0.05}O_{0.23}N_{0.03}C_{0.09}B_{0.13}$	4000
	$W_{0.21}Ti_{0.07}S_{0.22}Al_{0.05}O_{0.22}N_{0.03}C_{0.04}B_{0.16}$	4000
	$W_{0.2}Ti_{0.05}S_{0.18}Al_{0.09}O_{0.28}N_{0.03}C_{0.04}B_{0.13}$	7000
18	$W_{0.14}Ti_{0.05}S_{0.17}Al_{0.05}O_{0.24}N_{0.03}C_{0.17}B_{0.15}$	8000
	$W_{0.15}Ti_{0.07}S_{0.22}Al_{0.01}O_{0.3}N_{0.03}C_{0.04}B_{0.18}$	6000
	$W_{0.145}Ti_{0.05}S_{0.18}Al_{0.01}O_{0.365}N_{0.03}C_{0.04}B_{0.18}$	7500
19	$W_{0.14}Ti_{0.08}S_{0.21}Al_{0.01}O_{0.25}N_{0.03}C_{0.08}B_{0.2}$	8000
	$W_{0.13}Ti_{0.06}S_{0.15}Al_{0.01}O_{0.39}N_{0.03}C_{0.04}B_{0.19}$	6500

Compozitia probelor 9; 10, 12, realizate in faza 1 a proiectului - obtinuta prin RBS



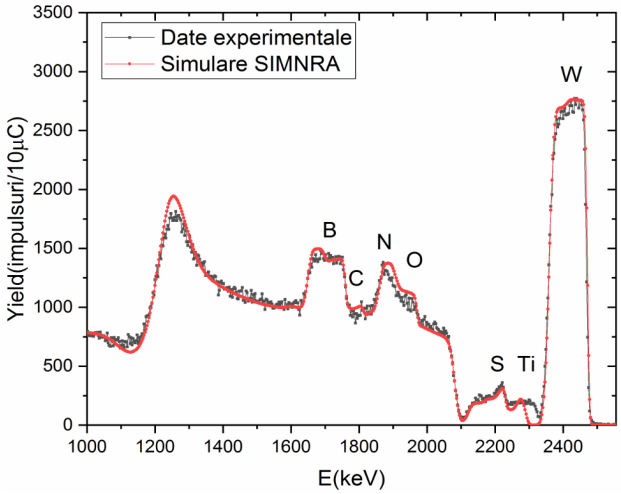
Proba 9

Strat unic cu compozitie constanta
B4C-60W + TiB2-90W + TiN-60W + WS2-100W



Proba 10

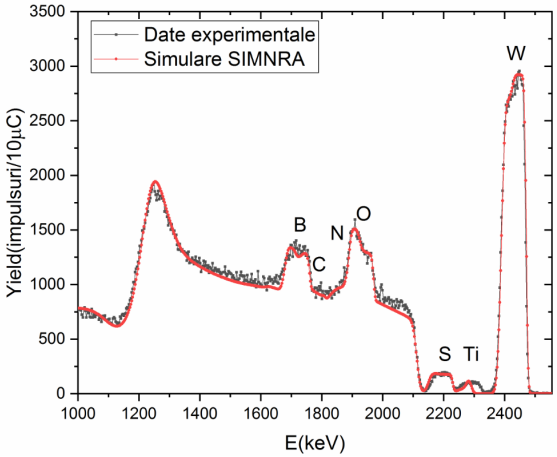
Multistrat -8 pachete cu compozitie modulata in pachet
[(B4C-24W + TiB2-240W + TiN-24W + WS2-100W) -5 min +
(B4C-36W + TiB2-180W + TiN-36W + WS2-100W) - 5 min +
(B4C-348W + TiB2-120W + TiN-72W + WS2-100W) – 5 min]



Proba 12

Strat unic cu compozitie constanta
B4C-36W + TiB2-180W + TiN-36W + WS2-100W

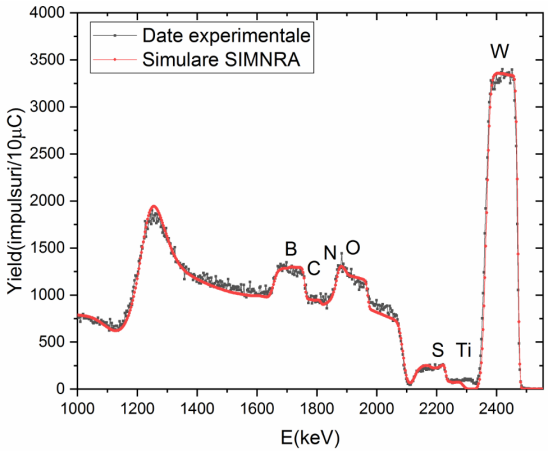
Compozitia probelor noi (15 – 19), realizate in faza 2 a proiectului - obtinuta prin RBS



Proba 15

Strat unic cu compozitie constanta

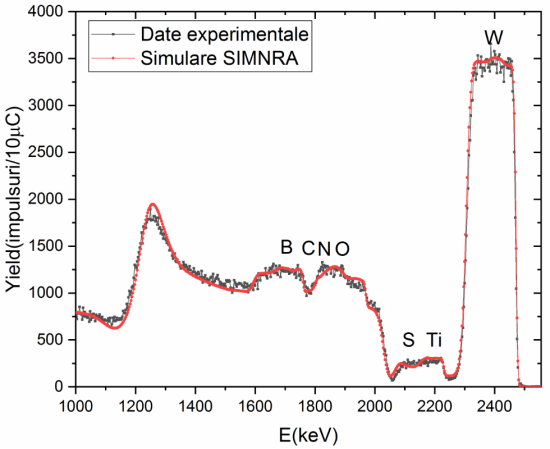
B4C-36W + TiB2-60W + TiN-36W + WS2-60W



Proba 16

Strat unic cu compozitie constanta

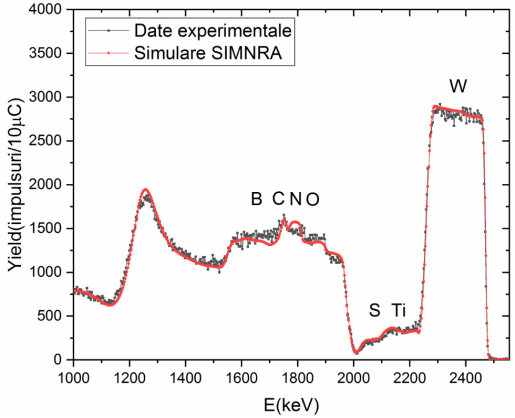
B4C-36W + TiB2-60W + TiN-36W + WS2-60W



Proba 17

Strat unic cu compozitie constanta

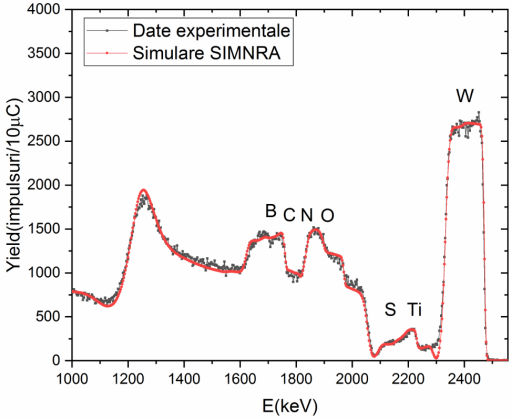
B4C-36W + **TiB**2-60W + **TiN**-36W + **WS2**-120W



Proba 18

Strat unic cu compozitie constanta

B4C-36W + **TiB**2-180W + **TiN**-36W + **WS2**-120W



Proba 19

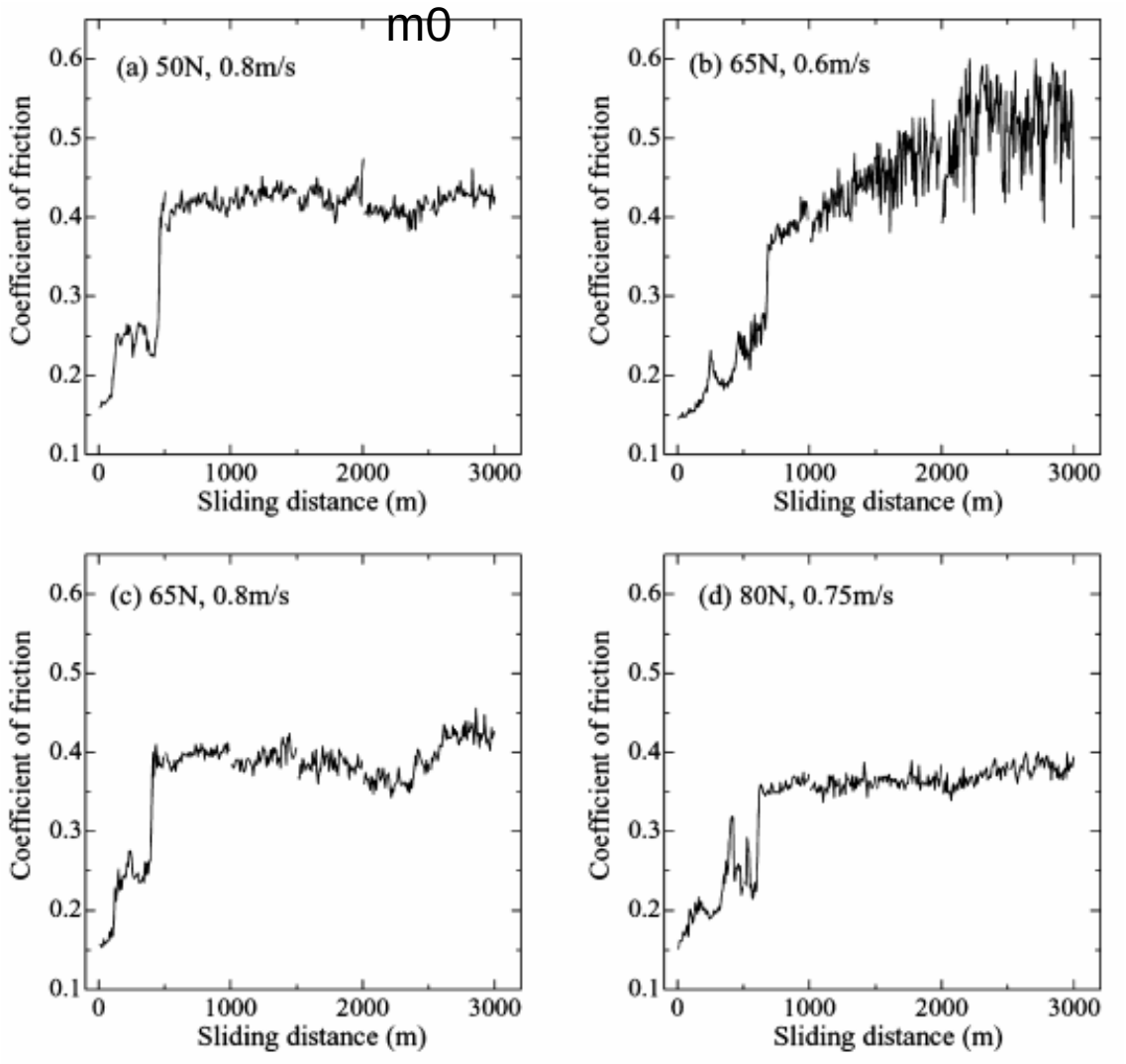
Multistrat din 12 pachete identice si repetitive, cu compozitie modulata in pachet

[(**B4C**-36W + **TiB**2-180W + **TiN**-36W + **WS2**-120W)/-5 min +
(**B4C**-36W + **TiB**2-120W + **TiN**-36W + **WS2**-80W) - 5 min +
(**B4C**-36W + **TiB**2-60W + **TiN**-36W + **WS2**-40W) – 5 min]

Compozitia probelor inainte si dupa optimizare - obtinuta prin SEM-EDS

Element chimic	*Substrat*	*Proba 9*	*Proba 10*	*Proba 12*	*Proba 15*	*Proba 16*	*Proba 17*	*Proba 18*	*Proba 19*	*Proba 20*
C	18,11	27,74	23,73	28,3	26,58	31,42	32,96	25,26	28,05	24,27
N		17,85		17,65	16,82	14,54	15,45	21,77	17,95	17,97
O	0,76	18,61	14,23	15,9	27,2	18,66	15,95	20,14	19,94	12,67
Al							0,95	0,3	0,47	0,44
Si	81,13		42,84							
S		16,85	8,25	17,4	13,23	16,66	16,65	14,55	15,7	20,25
Ti		6,5	5,7	8,65	4,04	3,89	2,8	6,13	6,44	10,2
W		12,45	5,25	12,1	12,13	14,83	15,24	11,85	11,45	13,02
Fe										1,18
Total	100	100	100	100	100	100	100	100	100	100

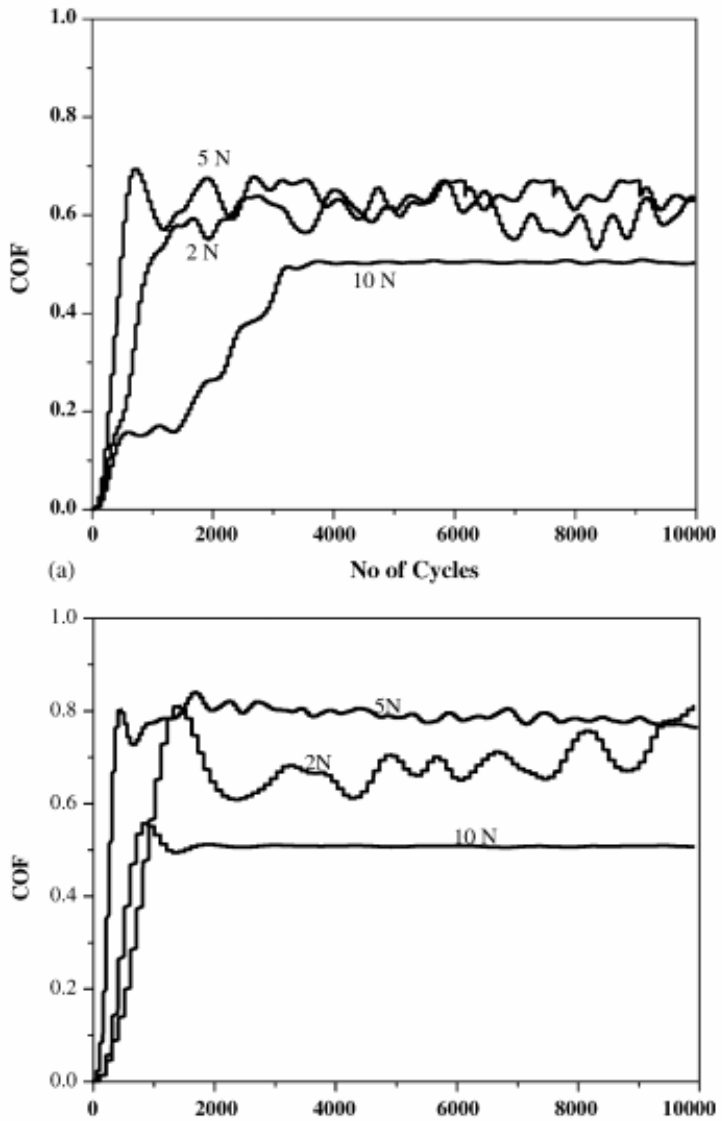
Dry sliding friction and wear properties of B_4C particulate-reinforced Al-5083 m0 matrix composite [1}



Rezultate publicate pentru CoF - B4C

Coefficient of friction (COF) with sliding distance for composite A under various testing conditions

Tribological properties of TiB_2 and TiB_2–$MoSi_2$ ceramic composites [2]0

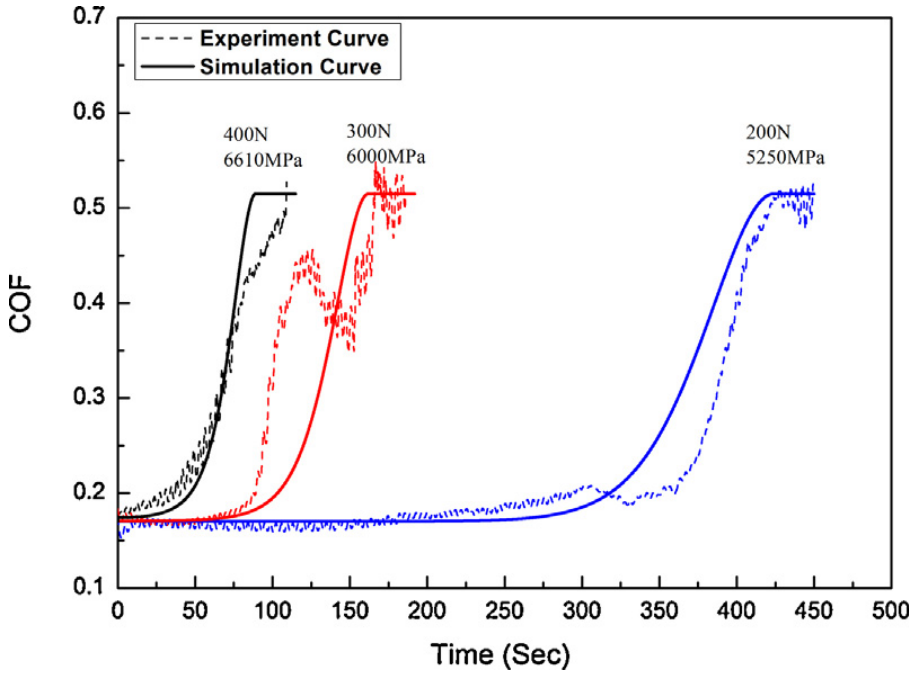


Rezultate publicate pentru
CoF – TiB2

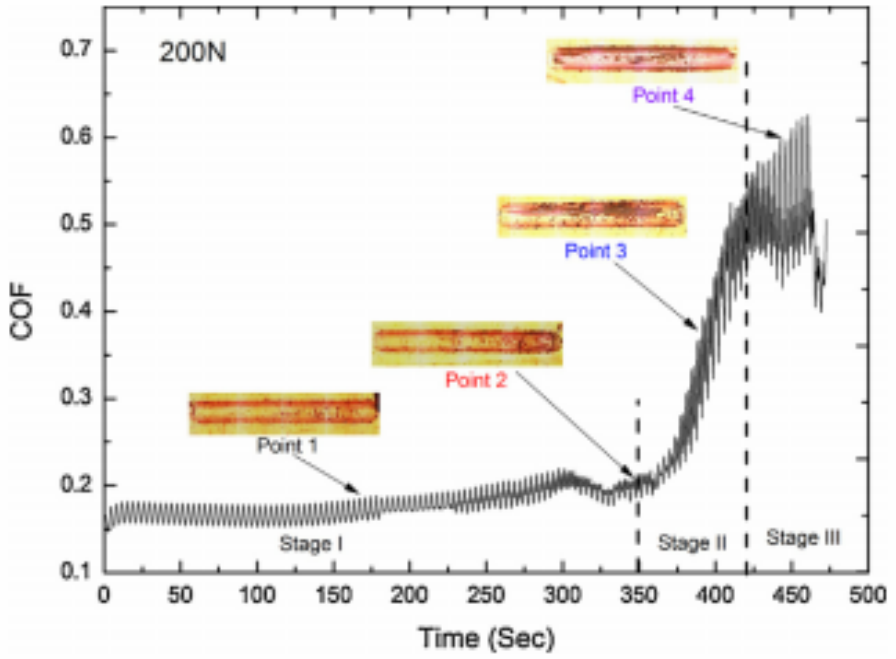
The frictional behavior of (a) monolithic TiB2 and (b) TiB2–MoSi2 (20 wt.%) composite, during fretting against bearing steel.

The friction coefficient evolution of a TiN coated contact during sliding wear [3]

Rezultate publicate pentru CoF - TiN



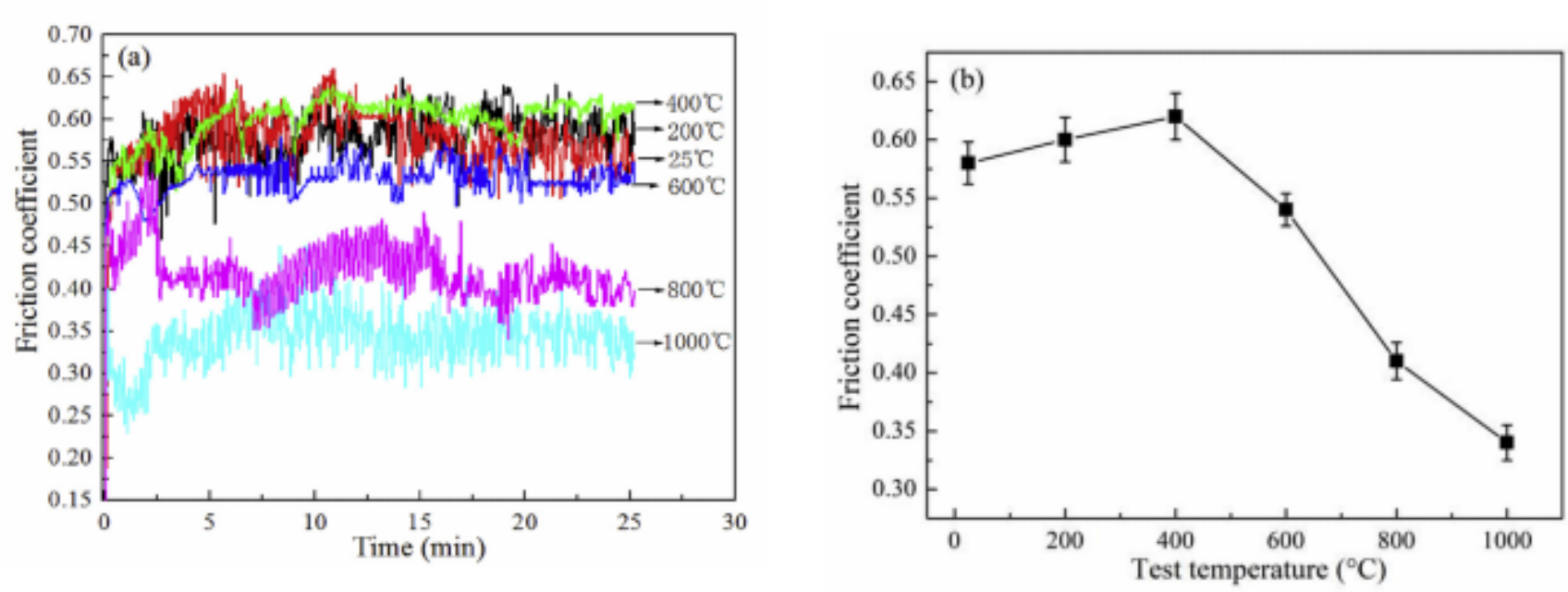
Simulative and experimental friction coefficient curves of the sample under 200 N, 300 N and 400 N.



Friction coefficient evolution of the sample under load of 200 N

Dry sliding wear behavior of $MoSi_2$-Mo_5Si-Mo_5SiB_2 composite at different temperatures and loads [4]

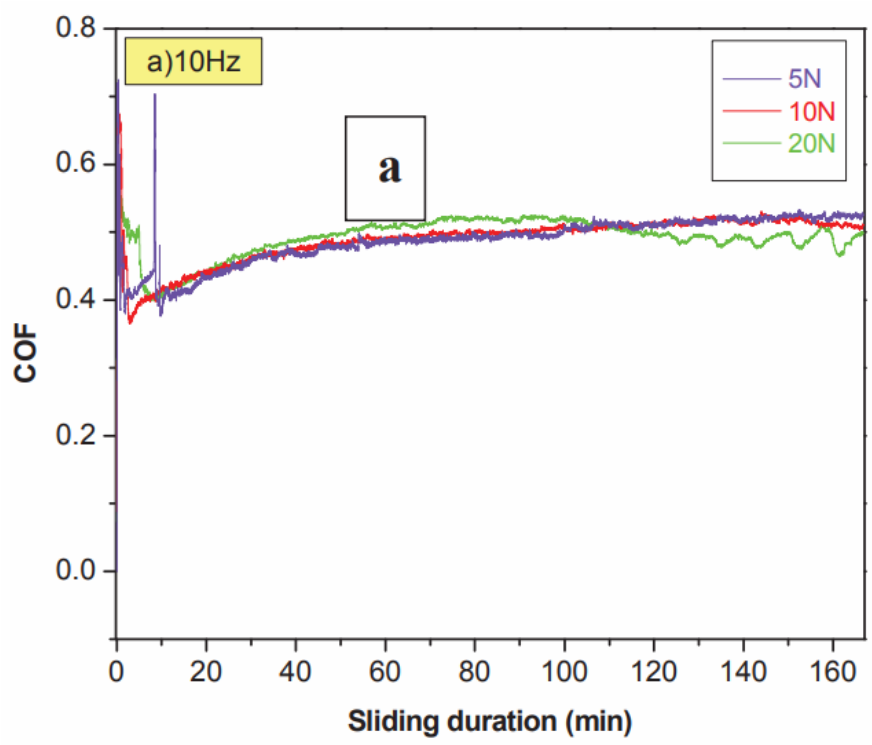
Rezultate publicate pentru CoF – MoSi2

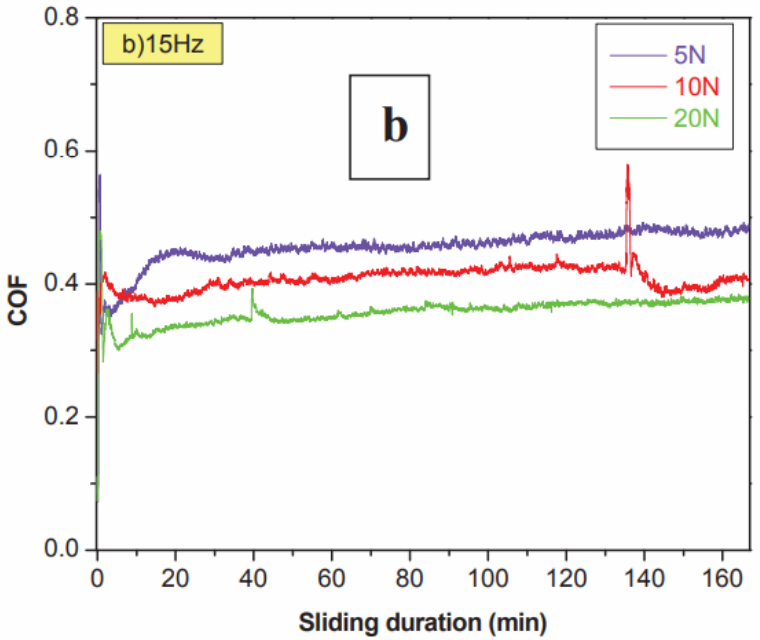


a Evolution of the friction coefficient versus time for sliding tests at different temperatures.
b Friction coefficient of the MoSi2-Mo5Si3-Mo5SiB2 composite at different test temperatures.

Tribology study on TiB_2+WSi_2 Composite against WC [5]

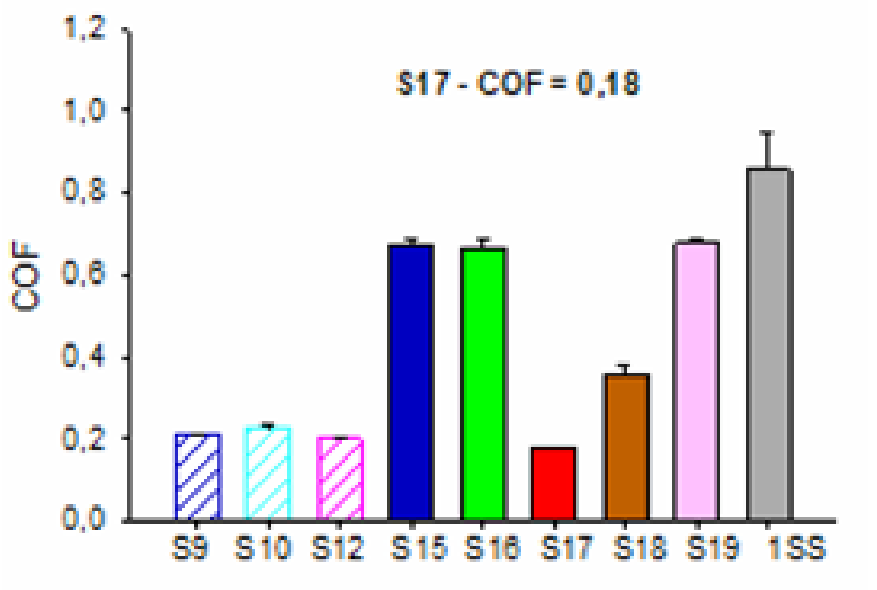
Rezultate publicate pentru CoF – TiB2 +WS2



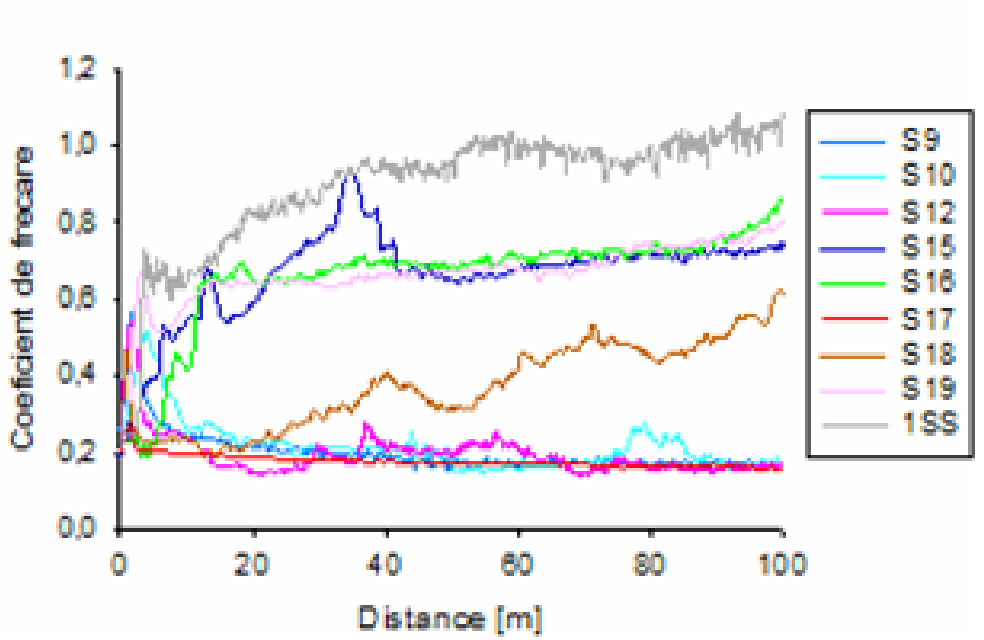


Rezultatele testului de Tribometrie *(Ball on Disck Tribometer Test)*

Pentru probele noi realizate –cu COF optimizat



a) Coeficientul mediu de frecare alunecare.



b) Variatia COF cu distanta de

Tab. 3. Rezultate brute/neprelucrate privind CoF si k (wear coefficient)
Proba 17 cu cel mai scazut COF(0,18) – din a 2-a grupa de materiale (B4C+TiB2+TiN +WS2

17

Tribo measurement1

Tribo parameters

Tribometer module / Version 4.4.K

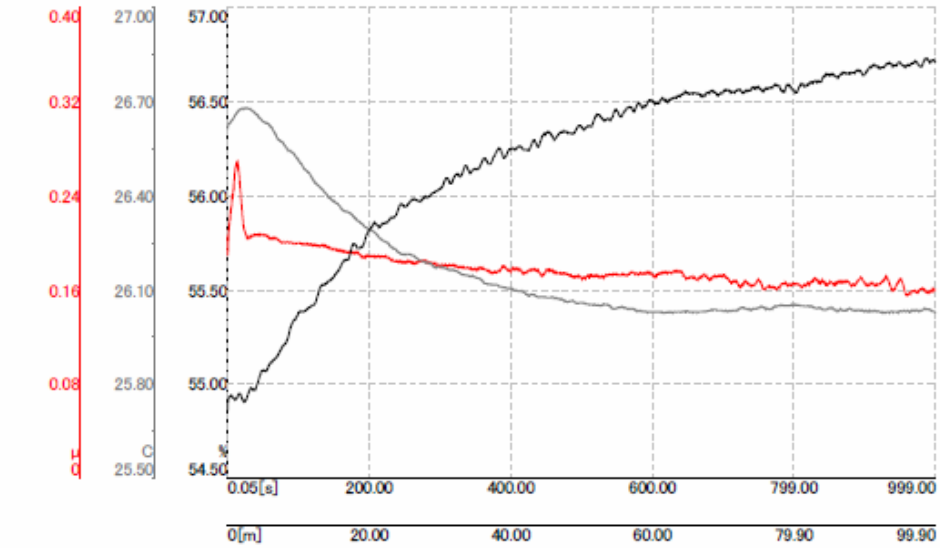
Acquisition
- Radius : 9.00 [mm]
- Lin. Speed : 10.00 [cm/s]
- Normal load : 2.00 [N]
- Stop condit. : 100.00 [m]
- Effective Stop : Meters
- Acquisition rate : 10.0 [hz]

Sample
- Cleaning : isopropanol

Static partner
- Coating : Al2O3
- Cleaning : isopropanol
- Dimension : 6.00 [mm]
- Geometry : Ball

Environment
- Temperature : 25.00 [<deg>C]
- Humidity : 49.00 [%]

Sample	Static partner	Calculations
Worn track section : 0.0 um2	Worn cap diameter : 0.0 um	Sample wear rate : 0 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 0 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa



Tab. 3. Rezultate brute/neprelucrate privind CoF si k (wear coefficient)
Proba 4 cu cel mai scazut COF (0,13) – din prima grupa de materiale (WC+TiB2+Ti+WS2)

4

Tribo measurement1

Tribo parameters

Tribometer module / Version 4.4.K

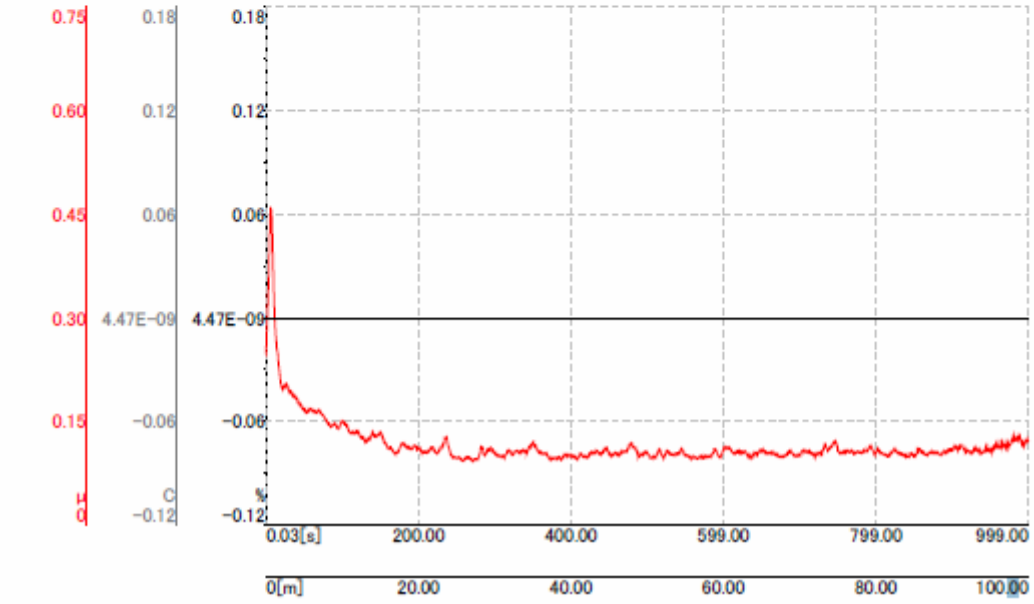
Acquisition
- Radius : 6.00 [mm]
- Lin. Speed : 10.00 [cm/s]
- Normal load : 1.00 [N]
- Stop condit. : 100.00 [m]
- Effective Stop : Meters
- Acquisition rate : 15.9 [hz]

Sample
- Cleaning : etanol

Static partner
- Coating : Al2O3
- Cleaning : etanol
- Dimension : 6.00 [mm]
- Geometry : Ball

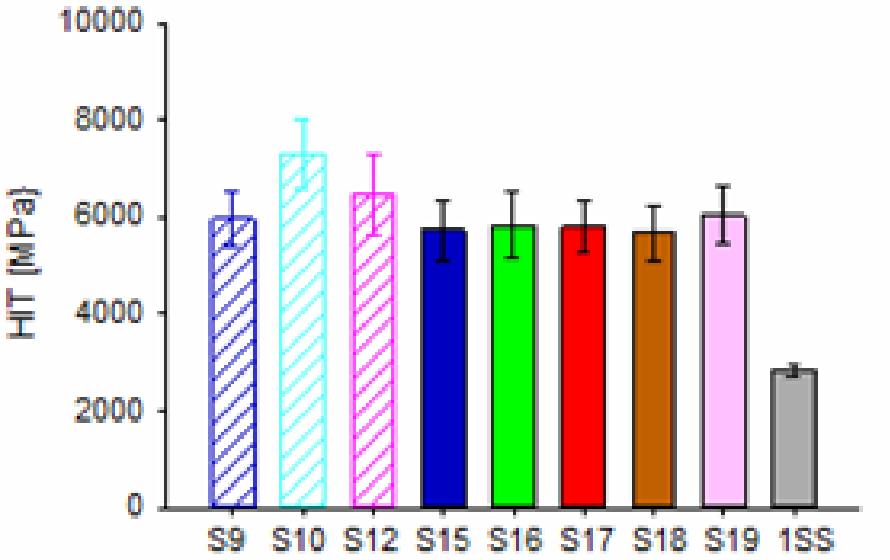
Environment
- Temperature : 0.00 [<deg>C]
- Humidity : 0.00 [%]

Sample	Static partner	Calculations
Worn track section : 0.0 um2	Worn cap diameter : 0.0 um	Sample wear rate : 0 mm3/n/m
Young's modulus : 0.0 gpa	Young's modulus : 0.0 gpa	Partner wear rate : 0 mm3/n/m
Poisson ratio : 0.000	Poisson ratio : 0.000	Max Herzian stress : 0 gpa

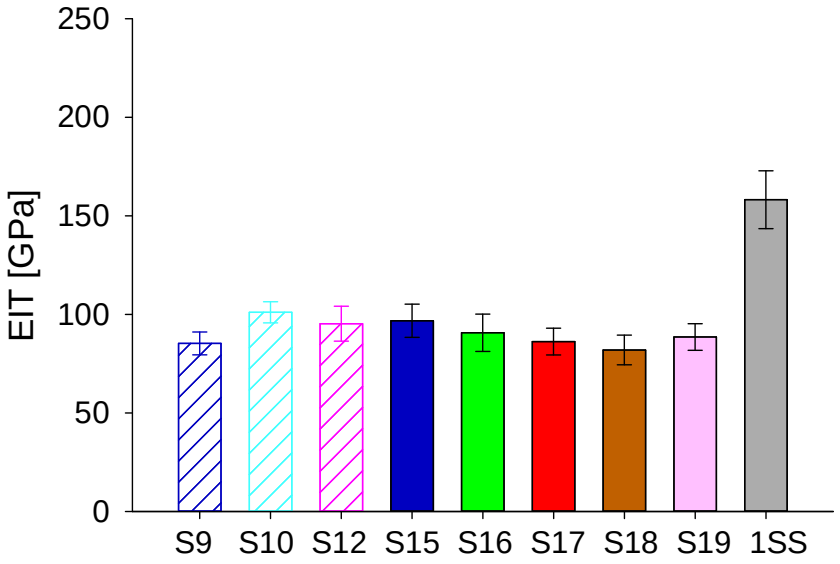


Rezultatele testului de duritate *(Hardness Test)*

Pentru probele noi realizate –cu COF optimizat



a) Valoarea duritatii



b) Valoare modulului de elasticitate

Rezultatele testului de duritate *(Hardness Test)*

Pentru proba nr 17 – cu cel mai scazut COF

17

Sample specifications

Reference :
Client :
Operator : DCR
Substrate :

Informations

Indentation # 1

Indentation parameters

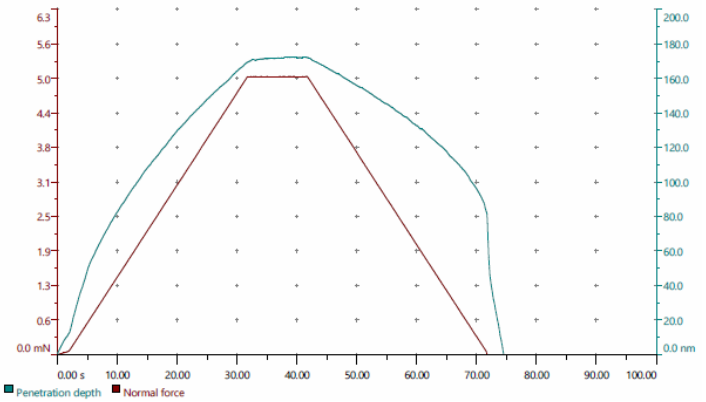
+ Standard
Acquisition Rate : 10.0 [Hz]
Linear Loading
Max load : 5.00 mN
Loading rate : 10.00 mN/min
Unloading rate : 10.00 mN/min
Pause : 10.0 s

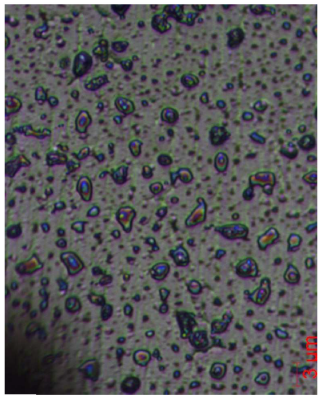
+ NHTX S/N: 01-04568 settings
Approach speed : 2000 nm/min
Retract speed : 2000 nm/min
Retract Time : 3 sec
Dz sensor in fine range
Delta Slope Contact : 80%
Date : 08-Jul-21
Hour : 3:22:59 PM

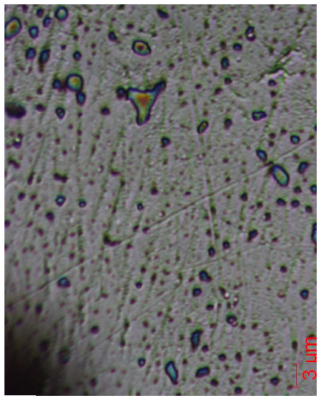
Indenters

Type : Berkovich
Serial number : CN-1
Material : Diamond

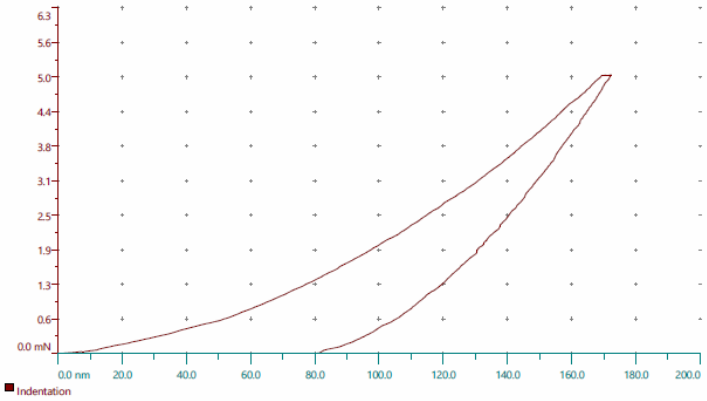
Curves







Indentation Images



Analysis # 1

Method
Oliver & Pharr

Main results
HIT= 5798 MPa
EIT= 81.961 GPa
E*= 90.067 GPa

Hypothesis
Poisson's ratio(nu)= 0.30

Additional results
HVIT= 536.96 Vickers
CIT 0.005/30/10= 1.65 %
nIT= 54.81 %
Fmax= 5.03 mN
hmax= 172.17 nm
S= 0.0908 mN/nm
hc= 131.19 nm
hr= 116.73 nm
hp= 79.22 nm
m= 1.6767
Epsilon= 0.74
R2= 1.000
Ap= 867754.59 nm²
Welast= 175.66 pJ
Wplast= 144.82 pJ
Wtotal= 320.47 pJ
Er= 83.508 GPa

Analysis # 2

Method
Martens hardness

Main results
HM= 3425.8 MPa

Additional results
CIT 0.005/30/10= 1.65 %
nIT= 54.81 %
Fmax= 5.03 mN
hmax= 172.10 nm
Ap= 1332789.31 nm²
Welast= 175.66 pJ
Wplast= 144.82 pJ
Wtotal= 320.47 pJ

Statistica datelor la testul de duritate (Hardness Test) – partea 1-a

		15	16	17	18	19
HIT (O&P)	Data : 1	6928.776	5797.582	5798.042	5420.832	5834.417
[MPa]	Data : 2	5419.073	5159.493	5486.436	5000.213	6367.399
	Data : 3	4773.193	4806.959	5951.313	5388.489	6494.236
	Data : 4	5443.678	5842.131	6370.230	5666.453	6888.484
	Data : 5	6033.405	5260.255	6107.837	5352.425	6343.804
	Data : 6	6081.206	6474.917	6610.511	5584.214	6825.204
	Data : 7	5736.549	7218.703	6141.016	5491.002	7225.184
	Data : 8	6011.028	6316.529	5175.025	5480.634	7228.535
	Data : 9	5287.740	6058.413	6064.756	6566.508	5224.821
	Data : 10	5450.057	5810.937	6158.654	5506.281	5374.924
	Data : 11	5626.665	6401.640	6179.570	6392.027	5452.860
	Data : 12	5070.754	5701.814	5696.662	6430.901	6097.946
	Data : 13	7154.441	4742.586	5657.814	6455.643	5122.140
	Data : 14	4809.594	4829.882	6357.025	6321.877	5606.043
	Data : 15	6110.943	6527.821	6286.145	4718.661	6211.552
	Data : 16	5746.630	5809.458	5880.636	4988.745	5623.886
	Data : 17	5139.019	5166.260	5465.496	4966.858	6120.126
	Data : 18	5367.092	6415.417	4829.630	5983.598	5412.541
	Data : 19	5683.855	6364.137	4841.036	6162.118	5799.729
	Data : 20	6415.765	5408.083	5895.465	5948.722	6164.953
	Data : 21	6345.469	6515.832	4810.556		5725.196
	Data : 22	5091.160				5840.175
	Data : 23	6135.421				
	Data : 24	5782.583				
	Mean	5735.170	5839.470	5798.279	5691.310	6044.734
	Std Dev	606.960	677.643	528.817	562.502	613.427

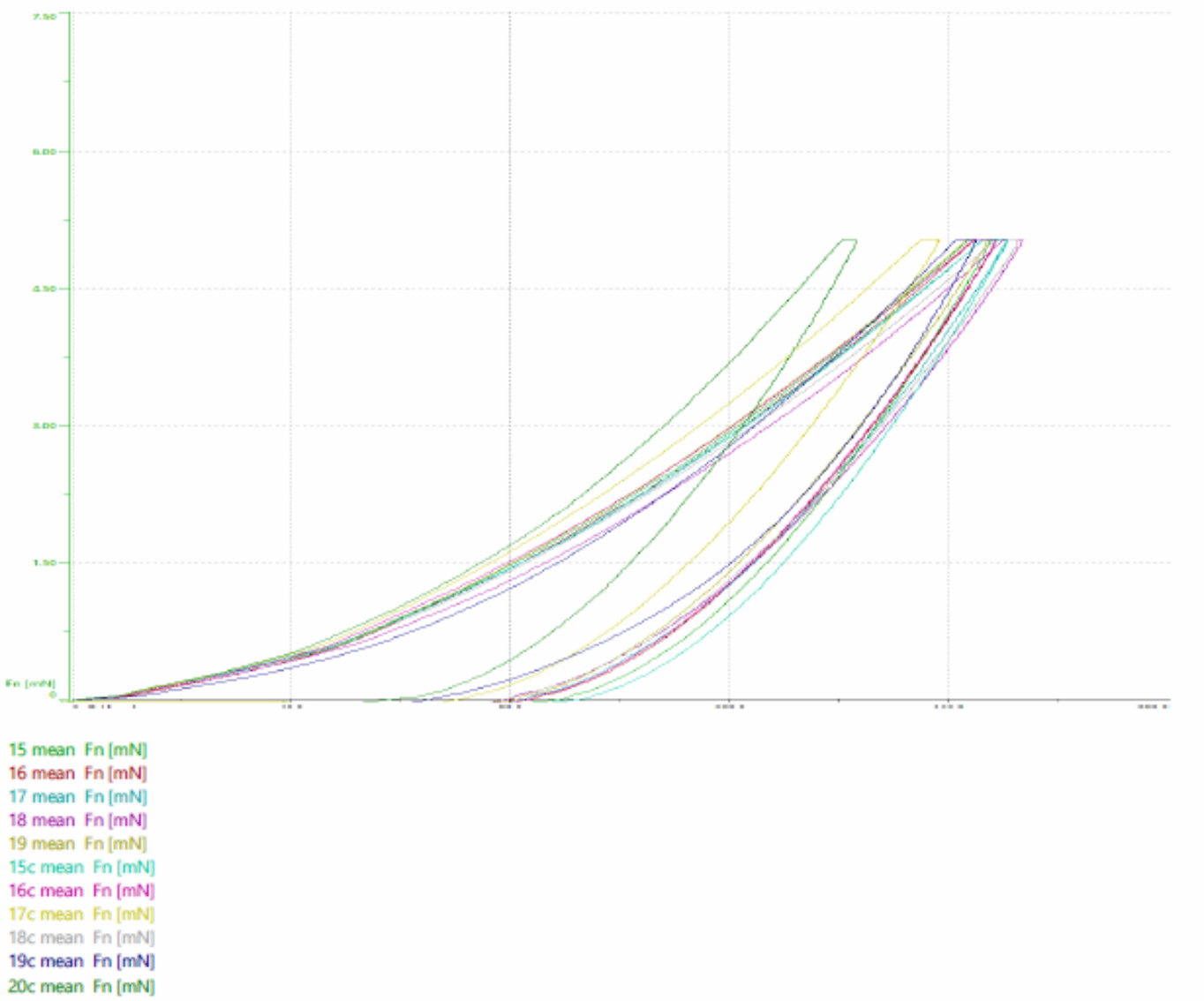
		15	16	17	18	19
EIT (O&P)	Data : 1	108.359	93.652	81.961	79.880	84.578
[GPa]	Data : 2	95.924	77.791	77.077	81.382	85.043
	Data : 3	85.325	80.508	78.991	85.935	89.104
	Data : 4	87.843	95.982	93.685	90.204	104.597
	Data : 5	92.815	96.370	85.781	78.752	89.935
	Data : 6	105.541	101.808	93.230	87.962	96.416
	Data : 7	98.632	100.340	85.363	80.269	101.369
	Data : 8	91.978	88.856	82.273	88.073	95.953
	Data : 9	97.856	111.218	82.038	85.162	83.514
	Data : 10	88.150	95.606	89.803	74.203	76.544
	Data : 11	91.513	96.147	84.140	85.046	81.898
	Data : 12	104.940	93.568	82.520	77.609	84.097
	Data : 13	94.286	77.405	86.898	93.385	88.232
	Data : 14	83.335	79.443	82.658	94.336	83.852
	Data : 15	98.557	85.643	82.584	69.720	92.383
	Data : 16	92.763	88.644	102.482	77.863	88.322
	Data : 17	86.712	79.910	102.107	64.897	93.290
	Data : 18	102.078	89.675	86.179	78.806	83.964
	Data : 19	94.927	98.551	79.051	88.290	85.591
	Data : 20	111.143	76.948	85.007	78.029	92.699
	Data : 21	99.683	97.456	87.895		85.517
	Data : 22	92.508				82.357
	Data : 23	116.722				
	Data : 24	101.886				
	Mean	96.811	90.739	86.272	81.990	88.602
	Std Dev	8.420	9.458	6.792	7.542	6.748

Statistica datelor la testul de duritate (Hardness Test) - partea a 2-a

		15	16	17	18	19
hm (O&P)	Data : 1	152.105	167.048	172.167	176.639	170.751
[nm]	Data : 2	171.336	182.393	178.455	181.638	165.584
	Data : 3	183.795	184.752	172.379	174.471	162.206
	Data : 4	173.831	165.614	161.037	169.398	152.641
	Data : 5	165.440	172.534	166.960	178.121	162.649
	Data : 6	160.188	157.529	158.909	171.231	156.331
	Data : 7	165.842	152.160	167.548	175.697	151.204
	Data : 8	166.746	164.877	178.971	172.582	153.833
	Data : 9	171.646	158.713	169.611	162.807	177.734
	Data : 10	173.906	166.522	164.835	179.474	180.618
	Data : 11	170.968	159.755	167.491	165.256	175.882
	Data : 12	172.685	167.874	172.268	168.915	168.554
	Data : 13	157.389	187.581	171.170	160.205	177.032
	Data : 14	184.328	185.006	167.030	161.016	174.589
	Data : 15	162.222	164.555	167.028	192.523	162.990
	Data : 16	168.270	169.311	163.097	183.627	171.138
	Data : 17	178.157	180.708	168.263	192.383	163.643
	Data : 18	169.344	161.840	182.436	171.598	175.488
	Data : 19	168.417	159.253	186.254	164.849	170.454
	Data : 20	155.112	179.959	170.350	172.444	163.494
	Data : 21	159.453	158.548	182.066		170.878
	Data : 22	176.800				172.023
	Data : 23	156.527				
	Data : 24	164.326				
	Mean	167.870	168.960	170.875	173.746	167.305
	Std Dev	8.540	10.505	7.202	9.170	8.509

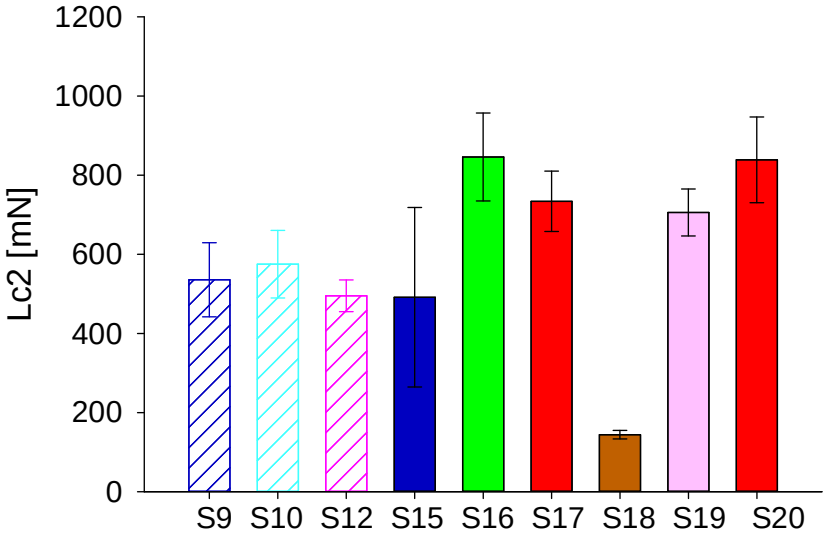
		15	16	17	18	19
Fm (O&P)	Data : 1	5.024	5.033	5.031	5.022	5.025
[mN]	Data : 2	5.038	5.024	5.031	5.028	5.037
	Data : 3	5.030	5.020	5.033	5.025	5.031
	Data : 4	5.022	5.035	5.035	5.023	5.021
	Data : 5	5.028	5.029	5.033	5.029	5.039
	Data : 6	5.027	5.025	5.027	5.023	5.033
	Data : 7	5.029	5.035	5.037	5.028	5.025
	Data : 8	5.030	5.029	5.034	5.030	5.029
	Data : 9	5.026	5.029	5.036	5.038	5.032
	Data : 10	5.028	5.037	5.032	5.030	5.028
	Data : 11	5.030	5.022	5.036	5.020	5.027
	Data : 12	5.025	5.027	5.023	5.021	5.019
	Data : 13	5.029	5.021	5.035	5.027	5.034
	Data : 14	5.030	5.026	5.034	5.023	5.026
	Data : 15	5.027	5.021	5.031	5.028	5.033
	Data : 16	5.025	5.038	5.032	5.038	5.034
	Data : 17	5.026	5.026	5.037	5.031	5.030
	Data : 18	5.026	5.035	5.034	5.024	5.037
	Data : 19	5.026	5.027	5.035	5.022	5.037
	Data : 20	5.031	5.025	5.028	5.023	5.036
	Data : 21	5.022	5.031	5.033		5.023
	Data : 22	5.028				5.030
	Data : 23	5.027				
	Data : 24	5.029				
	Mean	5.028	5.028	5.033	5.027	5.030
	Std Dev	0.003	0.005	0.003	0.005	0.005

Date de amprentare la testul de duritate (Hardness Test)

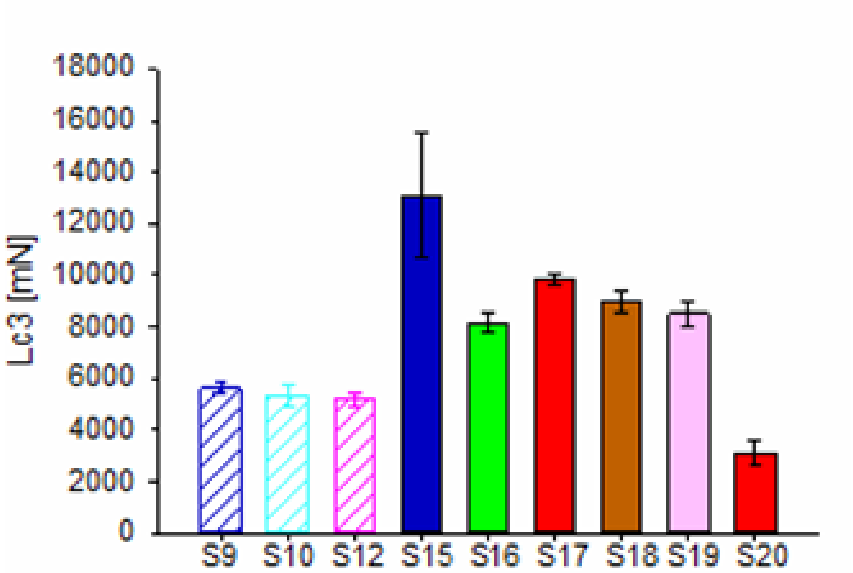


Rezultatele Testului de zgariere (Scratch Test)

Pentru probele noi realizate –cu COF optimizat



a) Valoarea sarcinii critice 2
(delaminare partiala)

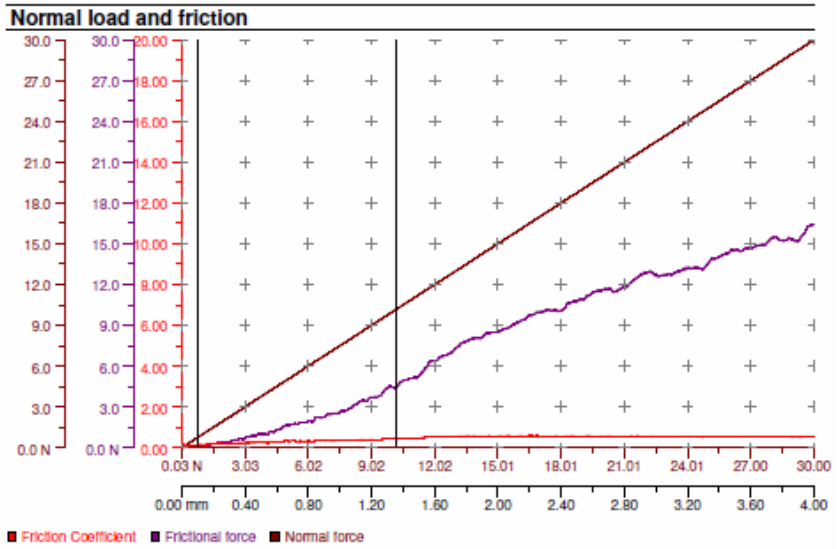


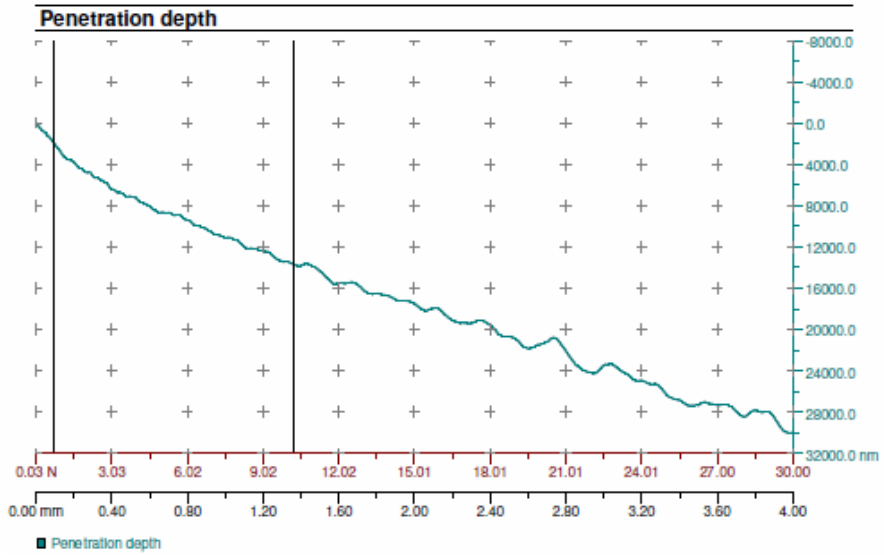
b) Valoarea sarcinii critice 3
(delaminare totala)

Rezultatele testului de Zgariere (ScratchTest) – Probele 15 - 19

		15	16	17	18	19
LC2 Optic	Data : 1	--,--	--,--	0.747	1.413	0.632
[N]	Data : 2	0.569	--,--	0.635	1.411	0.695
	Data : 3	0.646	--,--	0.660	1.291	--,--
	Data : 4	0.619	0.930	0.694	1.501	0.702
	Data : 5	0.462	0.720		1.405	0.798
	Data : 6	0.512	0.888		1.615	0.702
	Data : 7	0.537				
	Data : 8	--,--				
	Data : 9	0.633				
	Data : 10	--,--				
	Mean	0.568	0.846	0.734	1.439	0.706
	Std Dev	0.069	0.111	0.076	0.109	0.060
LC3 Optic	Data : 1	11.416	8.139	10.199	8.467	8.024
[N]	Data : 2	--,--	--,--	9.777	8.846	--,--
	Data : 3	--,--	--,--	9.786	9.114	--,--
	Data : 4	--,--	8.010	9.568	9.288	8.317
	Data : 5	--,--	7.853		8.755	8.490
	Data : 6	--,--	8.655		9.642	9.200
	Data : 7	--,--				
	Data : 8	14.797				
	Data : 9	--,--				
	Data : 10	--,--				
	Mean	13.106	8.164	9.833	9.019	8.508
	Std Dev	2.390	0.347	0.264	0.418	0.500

Rezultatele testului de Zgariere (ScratchTest) – Proba 17





17 : Scratch # 1

Scratch parameters

Linear Scratch	+ Instrument : MSTX S/N: 01-04568
Type : Progressive	+ Hardware settings
Begin Load (N) : 0.03	Fn contact : 0.03 N
End Load (N) : 30	Fn Speed : 5 N/s
Loading rate (N/min) : 14.98	Fn Remove speed : 10 N/s
	Approach speed : 2 %/s
AESensitivity : 8	Dz sensor in standard range
Speed (mm/min) : 2	
Length (mm) : 4	Date : 6/23/2021
Position X (mm) : 39.398	Hour : 10:04:16 AM
Position Y (mm) : 44.54	

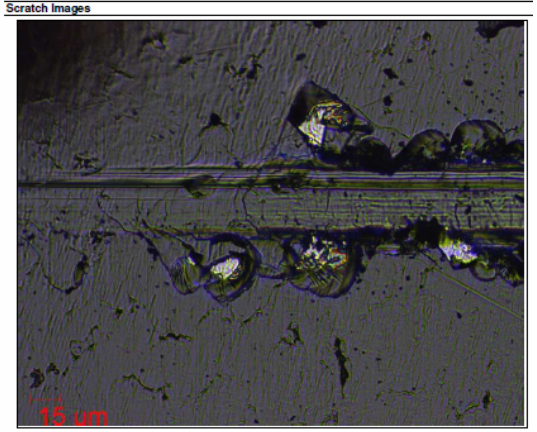
Indenters

Type : Rockwell	Material : 100C6
Serial number : I-184	Radius (µm) : 100

Critical loads

N	Optical		Ft	AE	Pd
Lc 1					
Lc 2	0.75	lc2			
Lc 3	10.2	lc3			
Lc 4					
Lc 5					

Scratch Images



Rezultatele testului de Zgariere (ScratchTest) – Proba 4

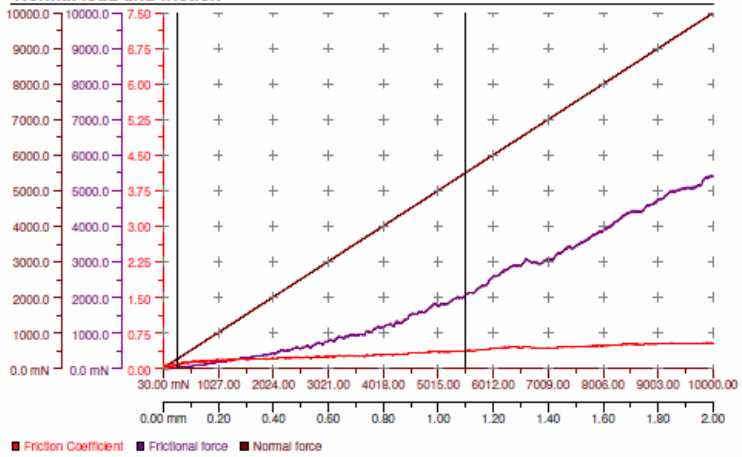
4

Sample specifications

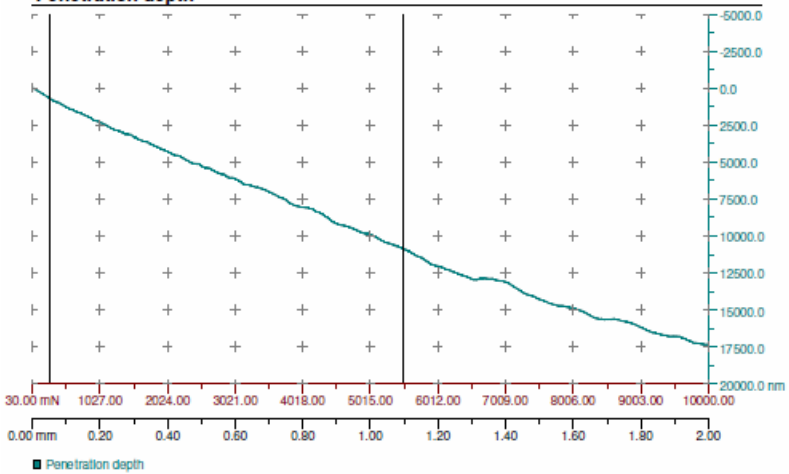
Reference :
Client : Mateescu
Operator : DCR
Substrate : SS

Layer	Coating	Process	Thickness
1	WC+TiB2+Ti+W..		0 nm

Normal load and friction



Penetration depth



4 : Scratch # 1

Scratch parameters

Linear Scratch
Type : Progressive
Begin Load (mN) : 30
End Load (mN) : 10000
Loading rate (mN/min) : 4985

AESensitivity : 8
Scanning load (mN) : 30
Speed (mm/min) : 1
Length (mm) : 2
Position X (mm) : 37.129

Position Y (mm) : 53.104
+ Instrument : MSTX S/N: 01-04568
+ Hardware settings
Fn contact : 30 mN
Fn Speed : 5000 mN/s
Fn Remove speed : 10000 mN/s
Approach speed : 2 %/s
Dz sensor in standard range

Date : 11/6/2020
Hour : 5:08:18 PM

Indenters

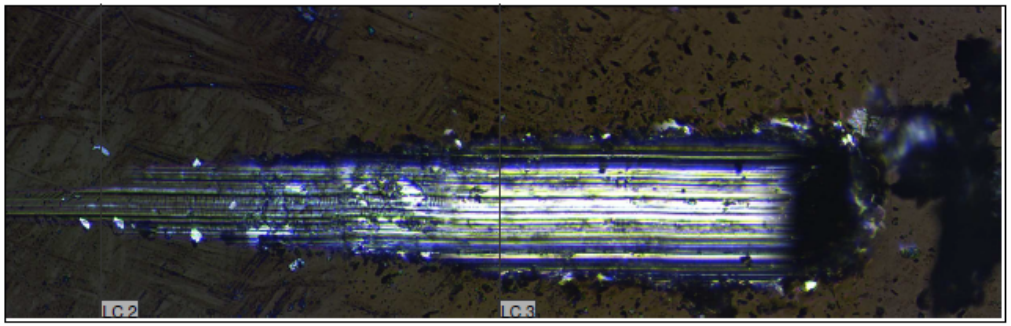
Type : Rockwell
Serial number : I-184

Material : 100C6
Radius (µm) : 100

Critical loads

mN	Optical		Ft	AE	Pd
Lc 1					
Lc 2	285.84	lc2			
Lc 3	#####	lc3			
Lc 4					
Lc 5					

Scratch Images



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Concluzii

In aceasta Faza s-au realizat straturi subtiri multicomponent din 2 Grupe Principale de materiale ***Ti, TiB_2, WC, WS_2 ,*** respectiv ***TiN, TiB_2, B_4C, WS_2*** in vederea obtinerii unor suprafete cu coeficient de frecare cat mai scazut

In Partea I a fazei s-a obtinut doar pentru Grupa Principala 1 de materiale un coeficent de frecare foarte scazut (0,147)

In Partea a II-a s-a urmarit obtinerea unui coefficient de frecare/CoF cat mai mic si pentru Grupa Principala 2 de materiale prin optimizarea parametrilor tehnologici de depunere si astfel s-a masurat o valoare medie de 0,180 la proba 17, pe intreaga durata a probei (lungime de alunecare

Duritatea cea mai ridicata s-a obtinut pentru suprafetele depuse cu materiale din Grupa Principala 2 (***B_4C)*** iar prin optimizare s-a urmarit imbunatatirea doar a COF si a coeficientului de uzura/k *(paramertii determinant pentru acoperirile tribologice)* nu si a duritatii.

Valorile COF si k pentru acoperirile tribologice pot fi previzionate sumar prin alegerea materialelor de depunere, dar valorile exacte ale acestora si comportarea in diverse conditii de temperatura, umiditate si sarcina de lucru, trebuie verificata.

MULȚUMESC