nano4ENERGY

DLC Coatings Deposited by Magnetron Sputtering on metallic and Insulating Substrates

Dr. Iván Fernández Martínez

Diamond Like Carbon (DLC)

Diamond-like Carbon (DLC) is a metastable form of amorphous carbon with significant sp3 bonding. DLC coatings are of interest in a wide variety of applications to provide low friction and high hardness and wear resistance.

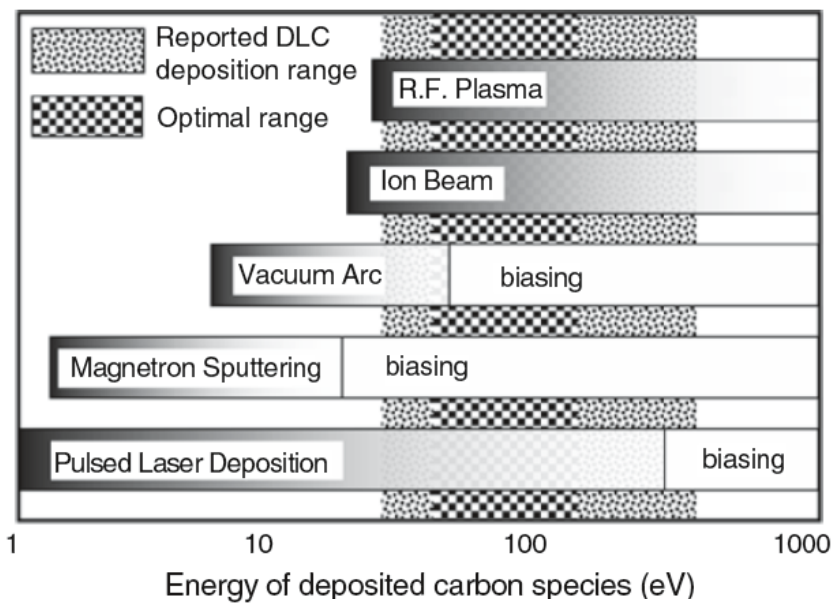
Applications

- **Glass: anti-scratch.**
- **Molds: prevent plastic transfer**
- **Engine parts: low friction (a-C).**
- **Space parts: low friction (a-C:H).**
- **Cutting tools: lubricated and dry conditions.**
- **Oil & gas: super lubricity in pipes.**
- **….**

DLC: State of the art

Material	Preparation technique	Density, (gm/cm^{-3})	% sp^3	Hardness, (GPa)	Young's modulus, (GPa)	Friction coefficient against metals*
Diamond	Naturally occuring	3.52	100	100	1,050	0.02–0.10
DLC	Sputtering	1.9–2.4	2–5	11–24	140	0.20–1.20
Me doped H:DLC	Reactive sputtering	–	–	10–20	100–200	0.10–0.20
H:DLC	r.f plasma	1.57–1.69	–	16–40	145	0.02–0.47
H:DLC, DLC	Ion beam	1.8–3.5	–	32–75	–	0.06–0.19
ta-DLC	Vacuum arc	2.8–3.0	85–95	40–100	500	0.04–0.14
ta-DLC	PLD	2.4	70–95	30–60	200–500	0.03–0.12

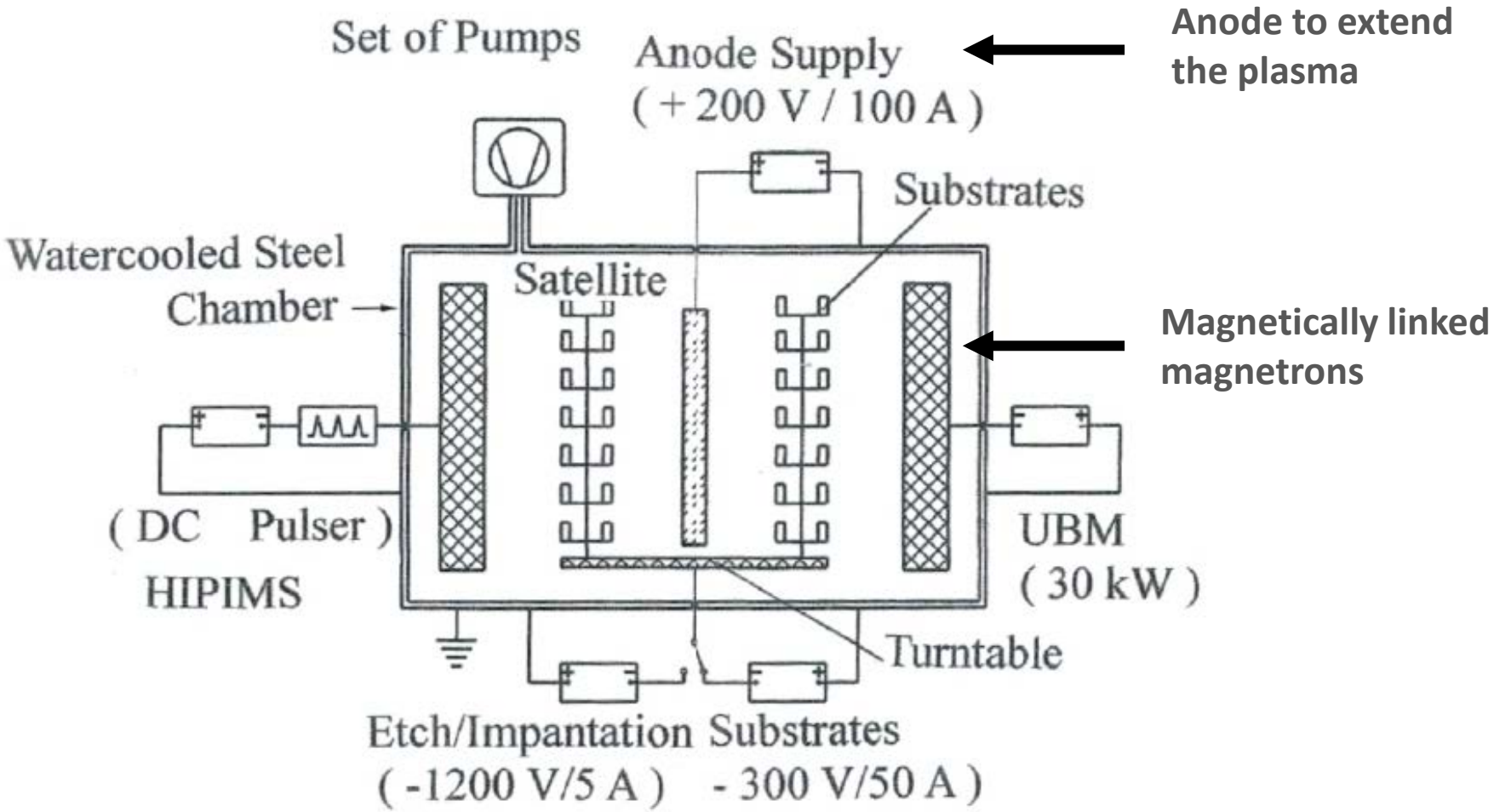
Ion assistance required ~ 100eV



A. Voevodin, Surf. Coat. Technol. 82 (1996) 199–213.

J. Robertson, Surf. Coat. Technol. 50 (1992) 185–203.

DLC hard coating magnetron deposition



Prof. Wolf-Dieter Munz "Industrial scale deposition of well adherent and low friction DLC coatings grown by HIPIMS and anode assisted unbalanced magnetron" – Private discussion

DLC hard coating magnetron deposition

55GPa Microhardess, comparable to filtered arc

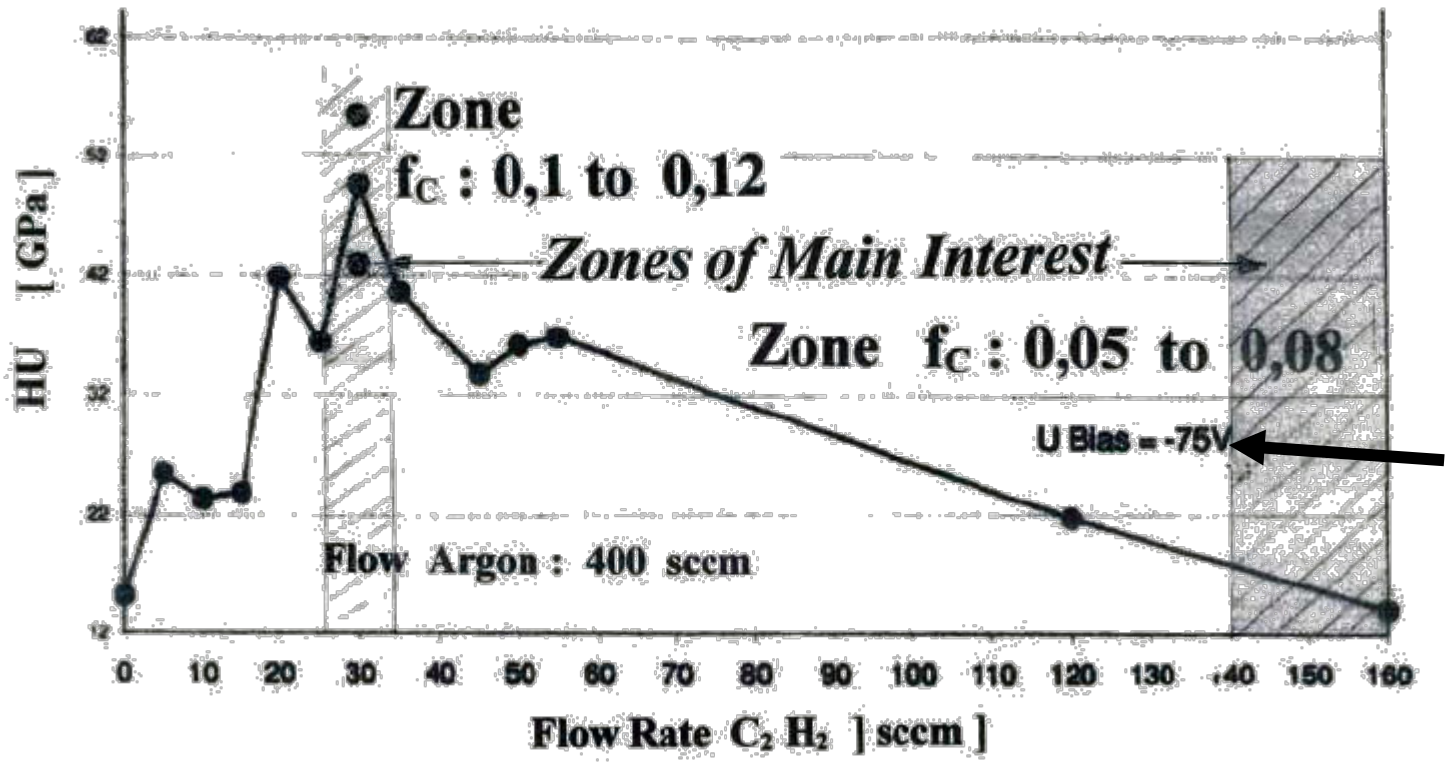


Fig. 7 Microhardness vs. Acethylene Flow

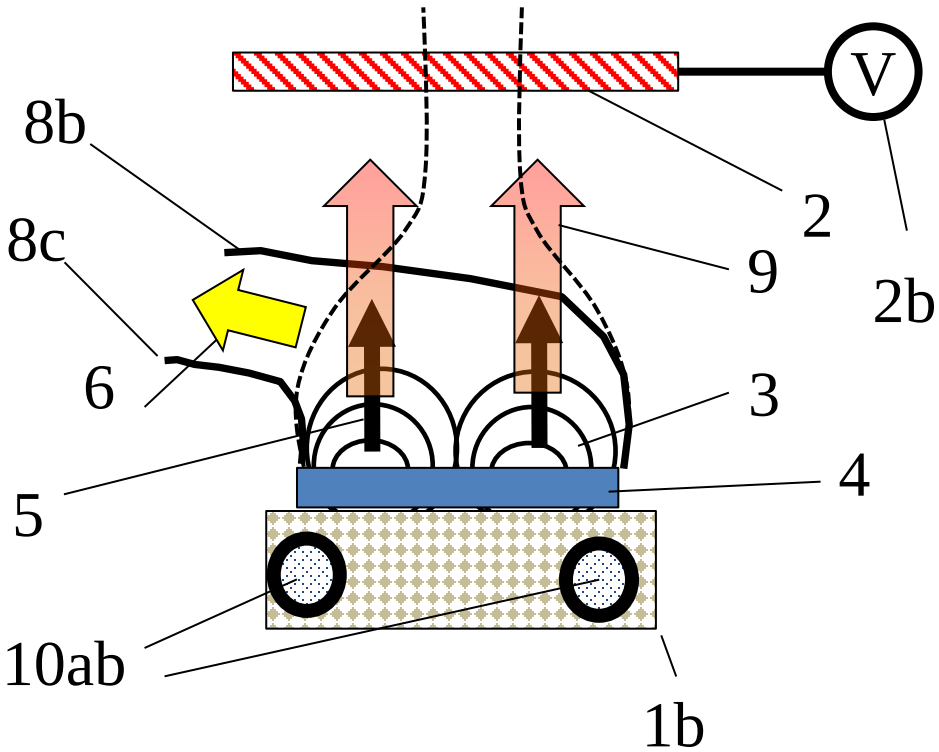
Bias required

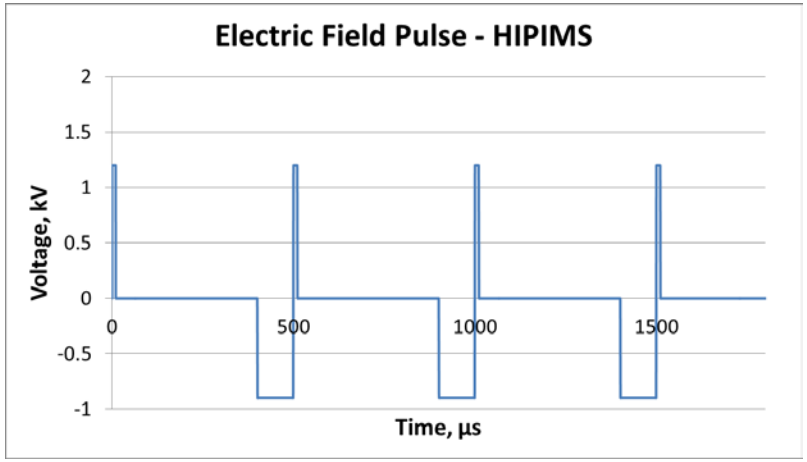
Glass?
Plastics?

Prof. Wolf-Dieter Munz "Industrial scale deposition of well adherent and low friction DLC coatings grown by HIPIMS and anode assisted unbalanced magnetron" – Private discussion

Ion assisted deposition on insulators?

Magnetron magnetic design (electron filter)
+ modified asymmetric bipolar pulsed PS



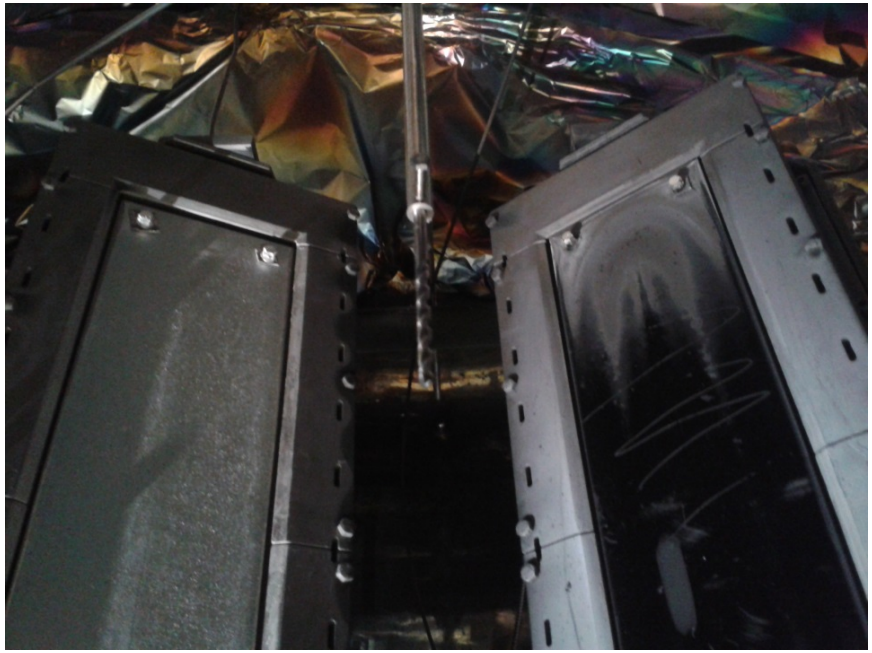


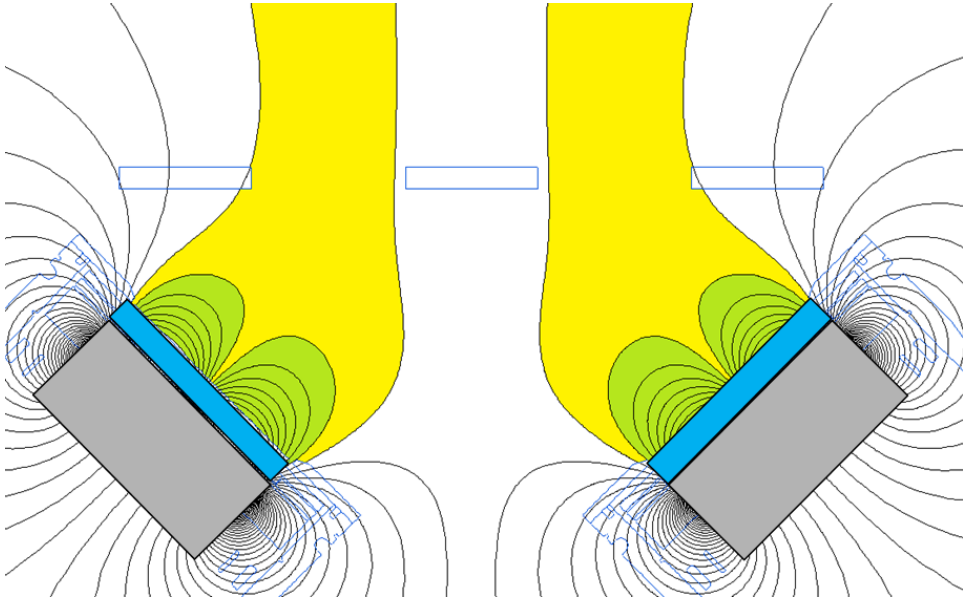
- **Positive ion bombardment**
- **No electron bombardent : Reduced thermal load?**

Proprietary technology: Patent application number GB1605162.5 (March 2016)

Ion assisted deposition on insulators?

Magnetron size 400x100mm2 (industrial relevant) – WC, Graphite targets
HIPIMS Ti/Cr + WC improves coating adhesion
Optimized magnetic field configuration to enhance ion assisted deposition





Proprietary technology: Patent application number GB1605162.5 (March 2016)

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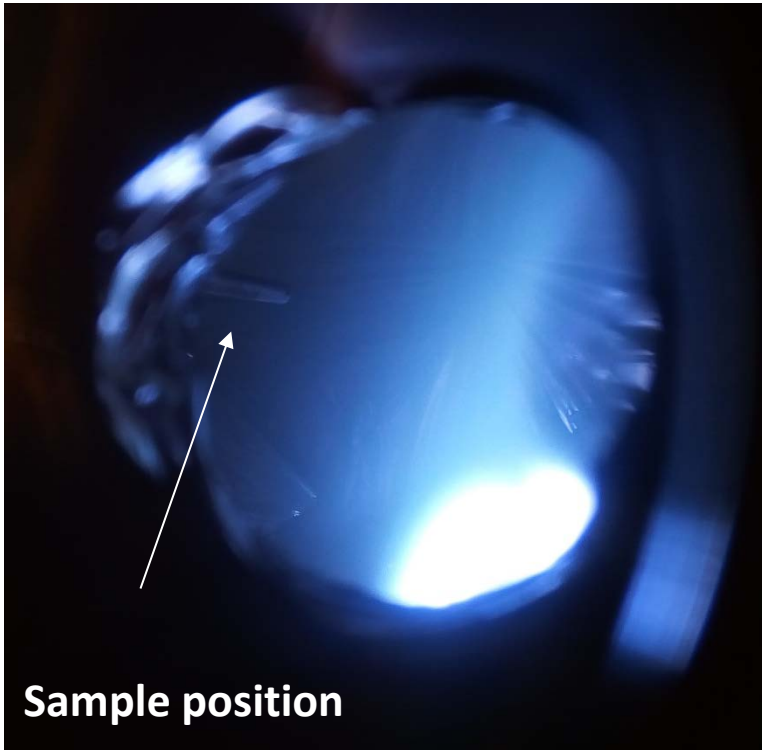

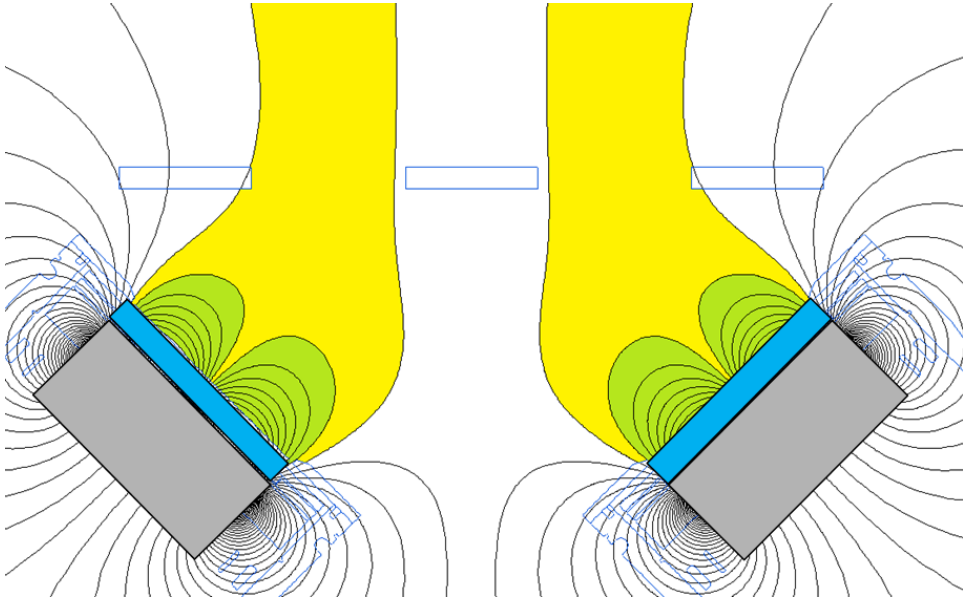

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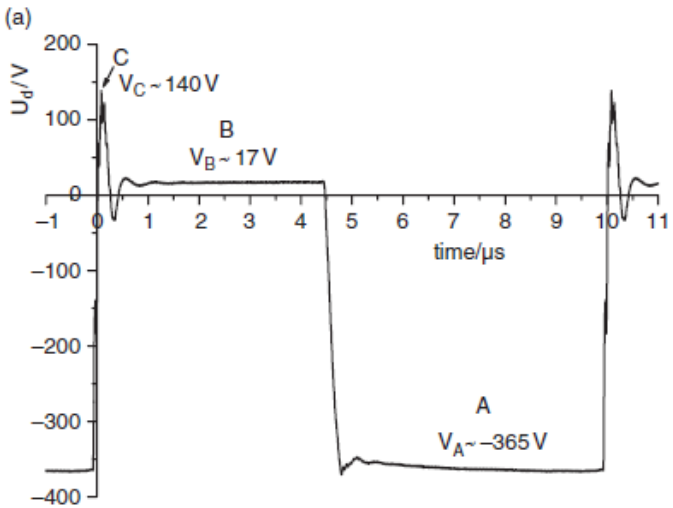


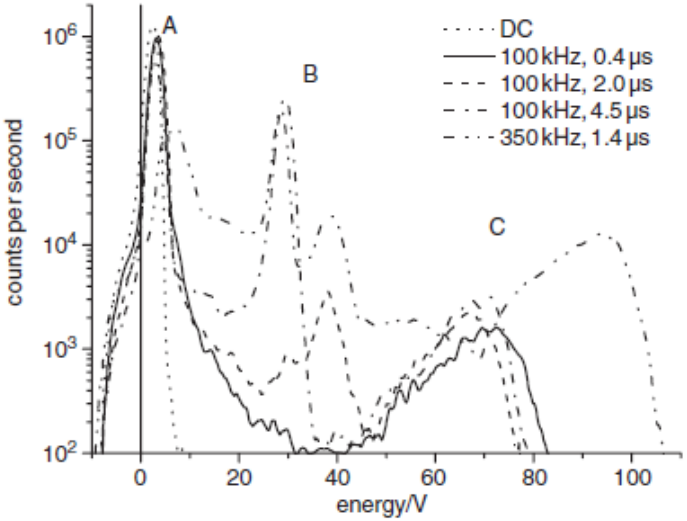


Figure 4. Ar^+ energy spectra (IEDF) for the cases: 100 kHz at 0.4, 2.0 and 4.5 μs reverse time and 350 kHz at 1.4 μs reverse time.

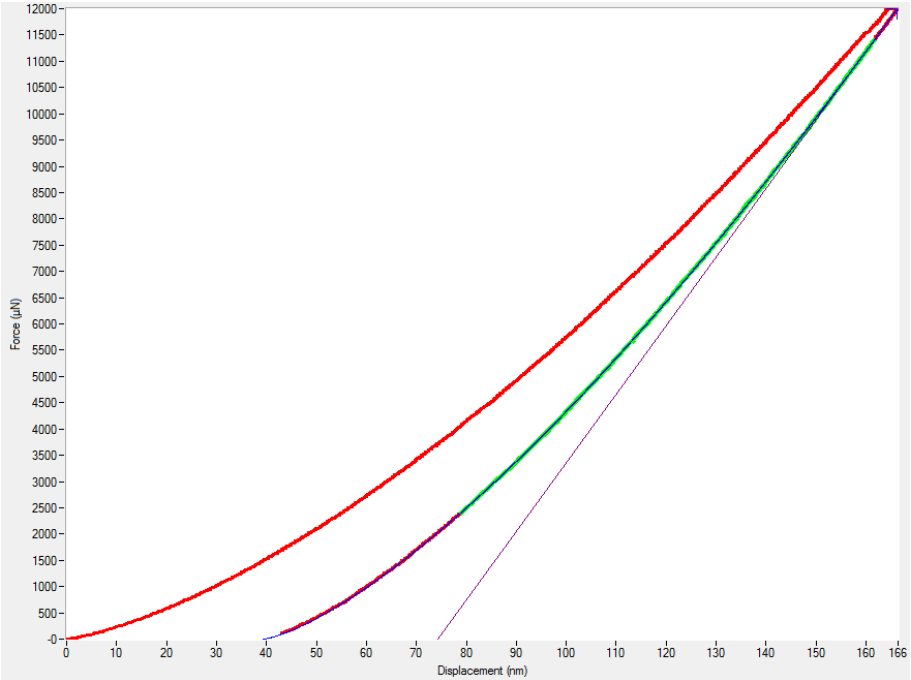
INSTITUTE OF PHYSICS PUBLISHING
PLASMA SOURCES SCIENCE AND TECHNOLOGY
Plasma Sources Sci. Technol. **11** (2002) 165–174
PII: S0963-0252(02)34675-9

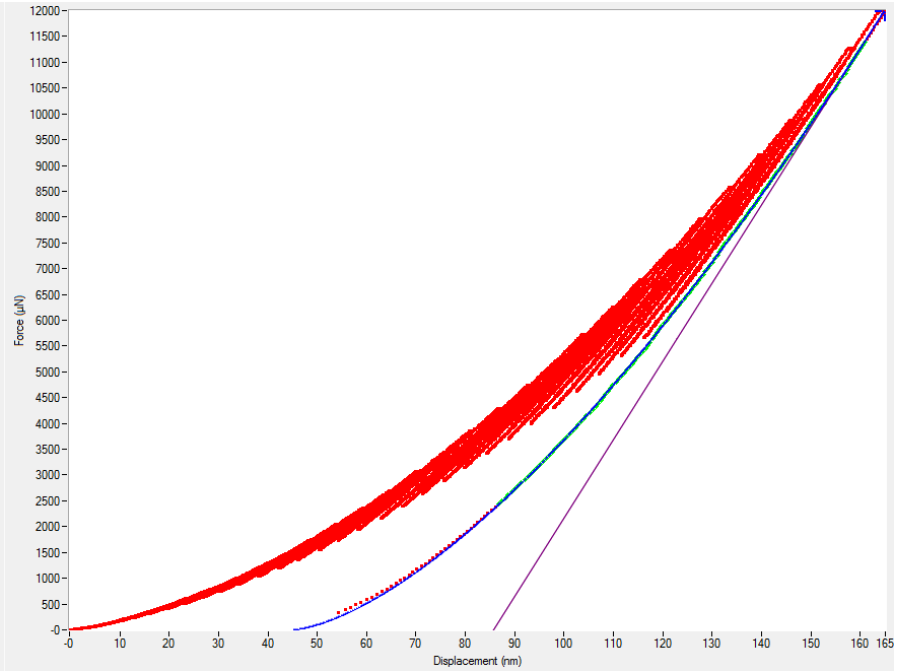
The distribution of ion energies at the substrate in an asymmetric bi-polar pulsed DC magnetron discharge

J W Bradley[1], H Bäcker[1], Y Aranda-Gonzalvo[1], P J Kelly[2] and R D Arnell[2]

3 energy regimes are measured

Coating Hardness: above 30GPa on floating substrate





Berkovich indenter
Single indents: 2,7, 12 mN
Load-partial Unload: 12 mN
Hysitron TI950

Hardness = 32 Gpa
Young modulus = 220 Gpa
500nm thick DLC

DLC hard coating magnetron deposition

15GPa Microhardess with -75V Bias!

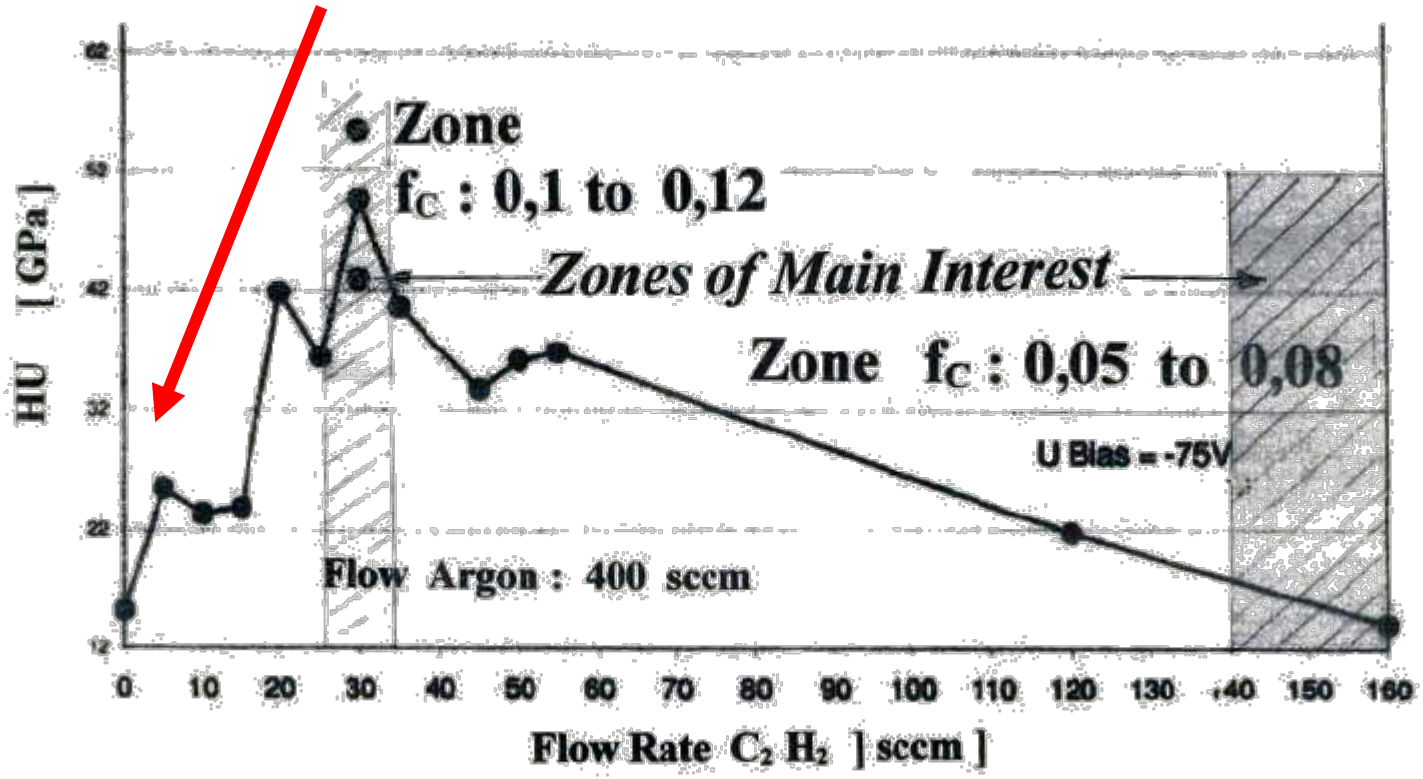


Fig. 7 Microhardness vs. Acethylene Flow

Bias required

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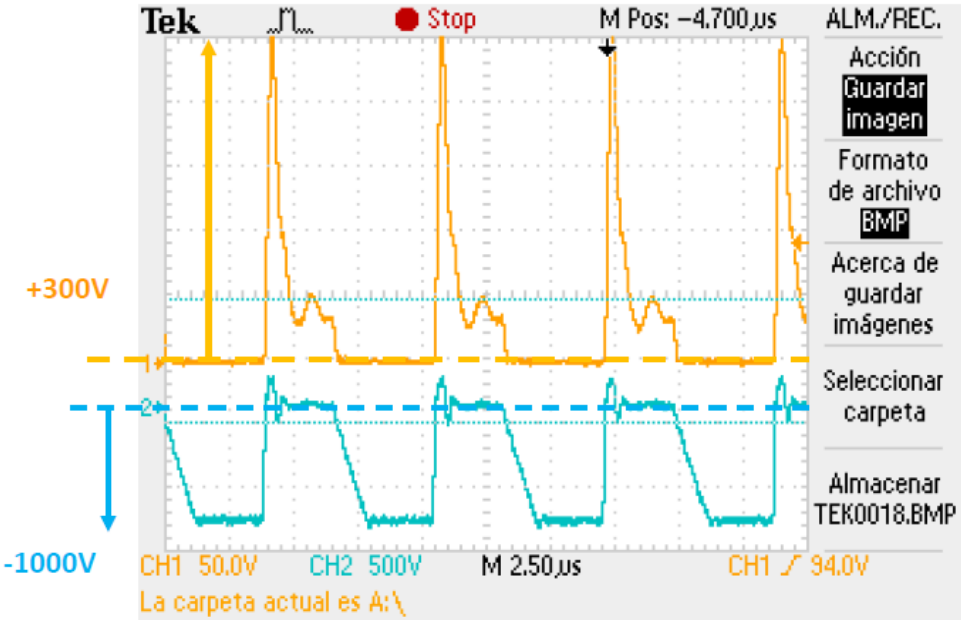
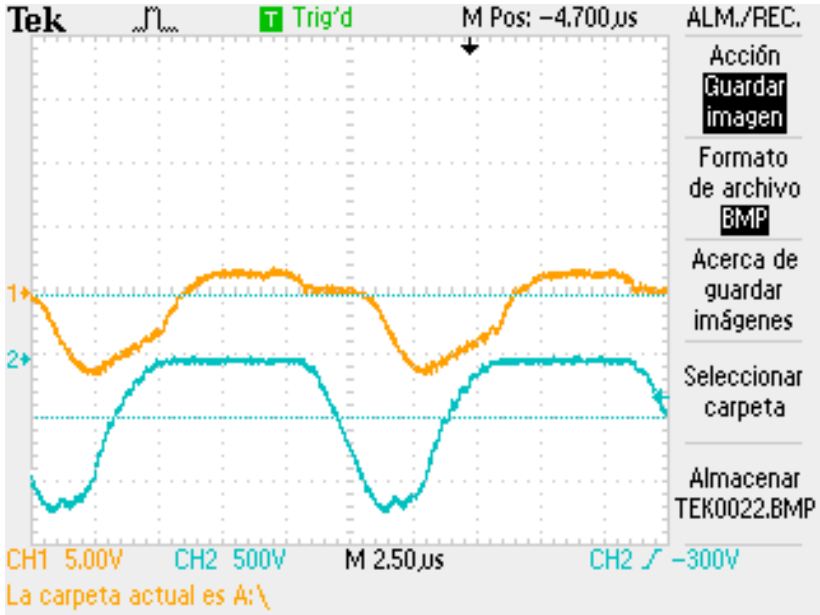
Ion assisted deposition on insulators?



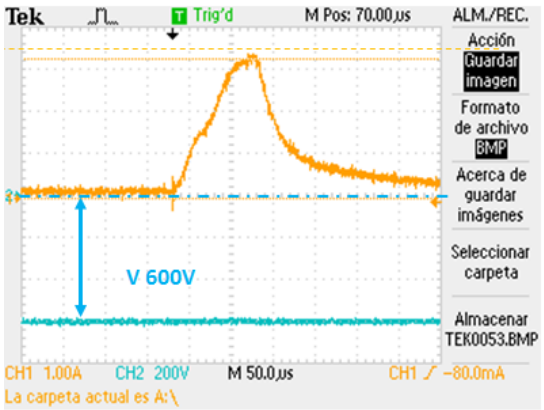


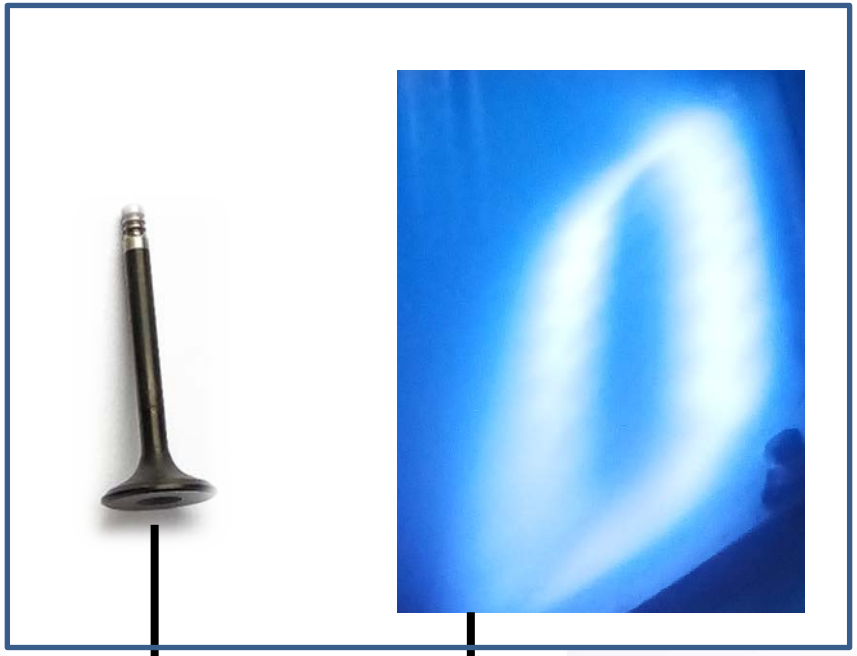
Hardness > 30GPa | **Hardness < 12GPa**

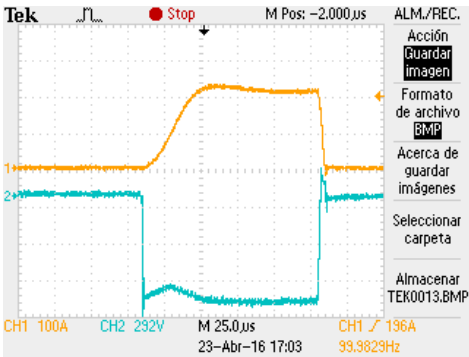
Modified DC-Pulsed PS | **Commercial DC-Pulsed PS**

Proprietary technology: Patent application number GB1605162.5 (March 2016)

Metallic substrates : HIPIMS implantation to promote coating adhesion

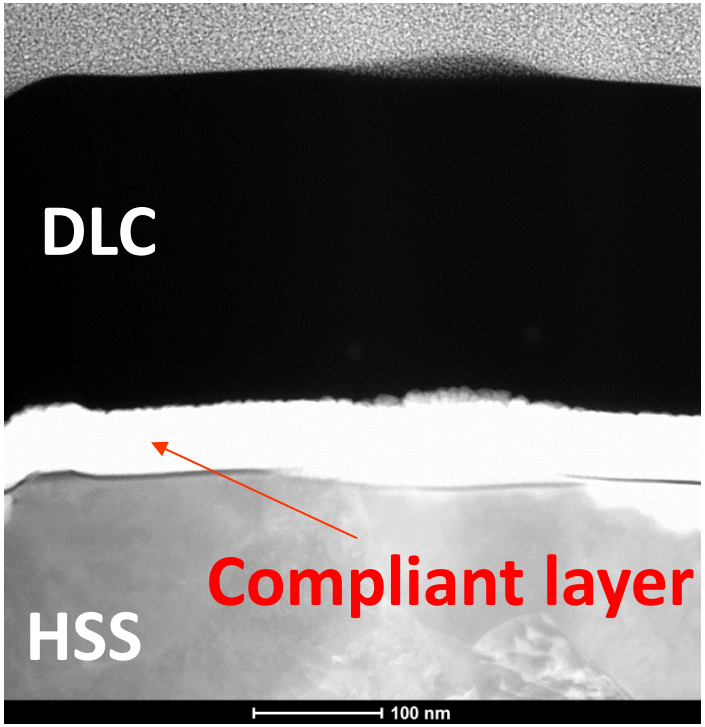

Tek
Trig'd
M Pos: 70.00µs
ALM./REC.
Acción
Guardar imagen
Formato de archivo
BMP
Acerca de guardar imágenes
Seleccionar carpeta
Almacenar TEK0053.BMP
V 600V
CH1 1.00A
CH2 200V
M 50.0µs
CH1 ∕ −80.0mA
La carpeta actual es A:\

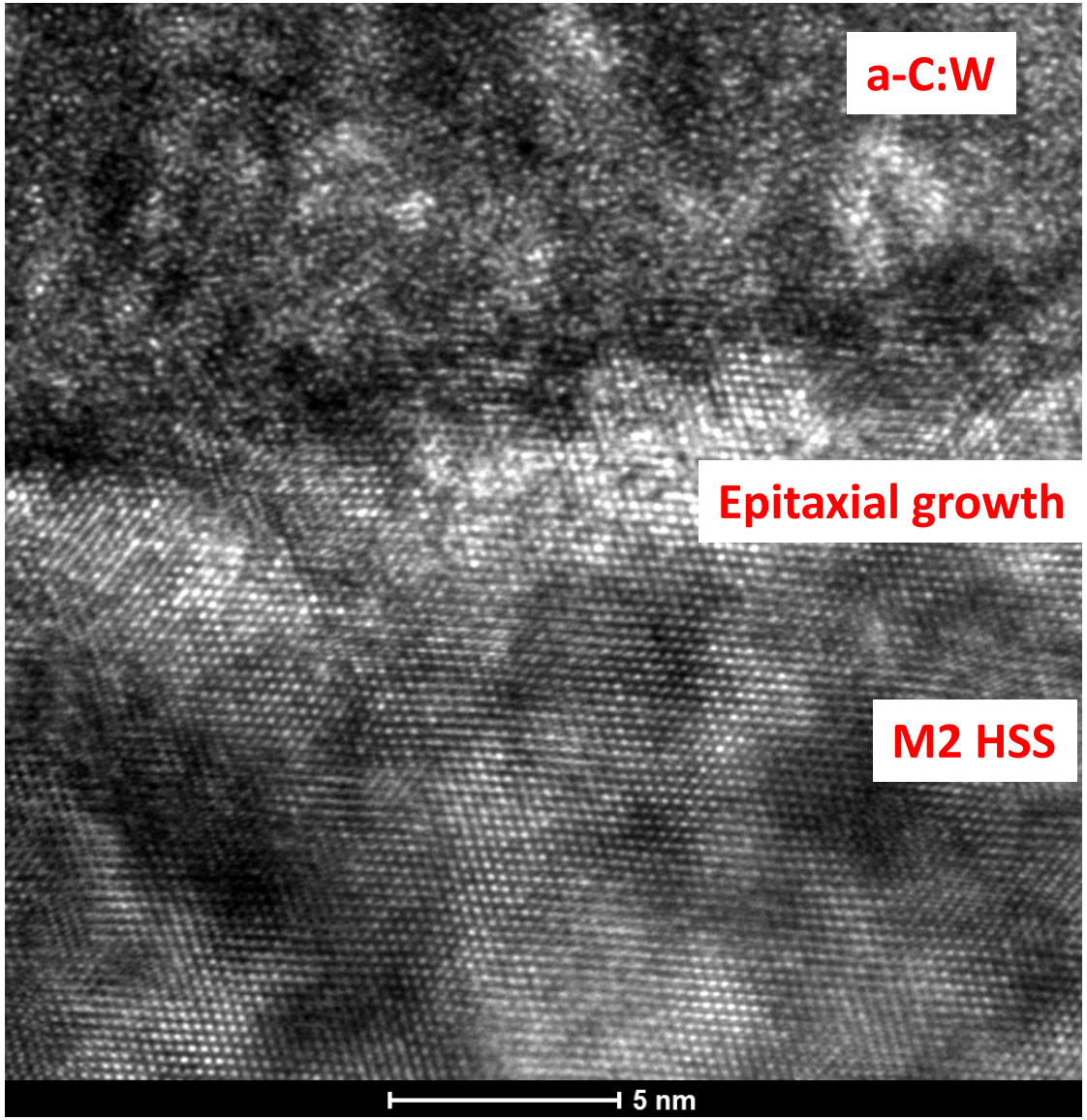


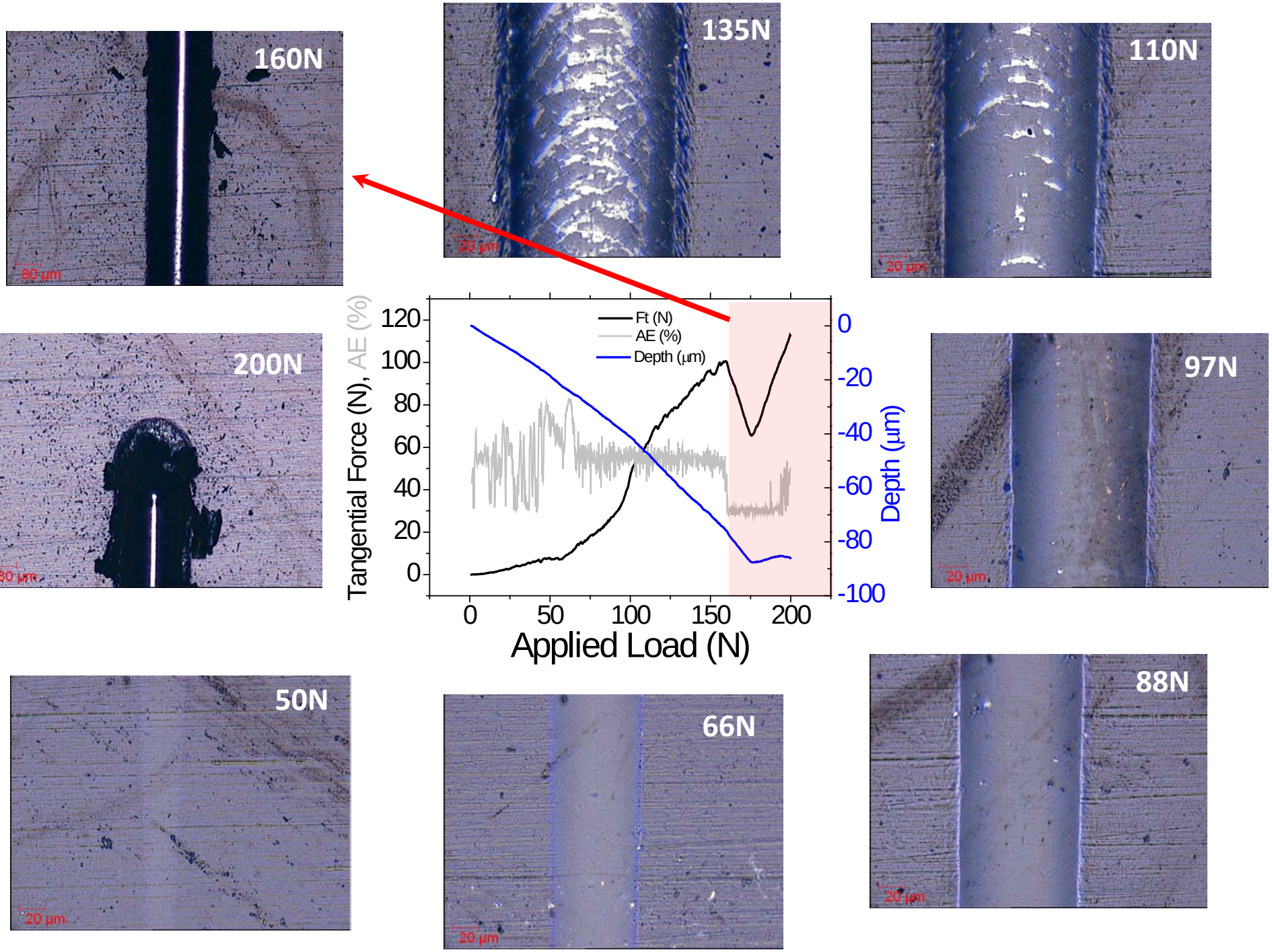

Tek
Stop
M Pos: −2.000µs
ALM./REC.
Acción
Guardar imagen
Formato de archivo
BMP
Acerca de guardar imágenes
Seleccionar carpeta
Almacenar TEK0013.BMP
CH1 100A
CH2 292V
M 25.0µs
23-Abr-16 17:03
CH1 ∕ 196A
99.9829Hz

Bias

HIPIMS


DLC
Compliant layer
HSS
100 nm

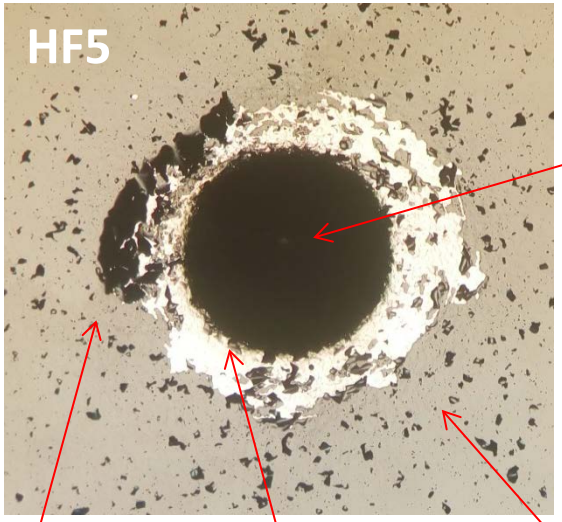

a-C:W
Epitaxial growth
M2 HSS
5 nm

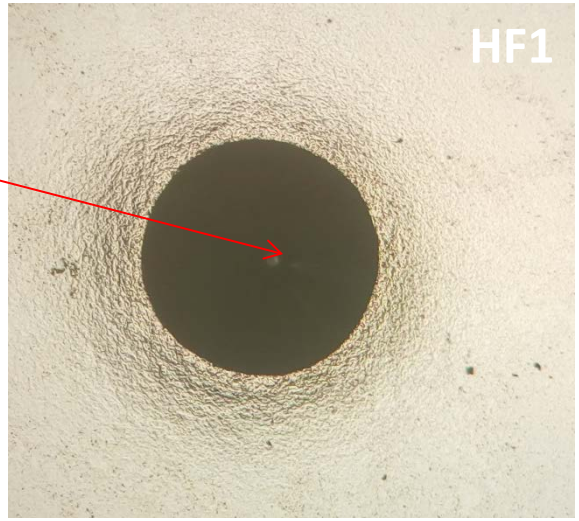

160N
135N
110N
200N
97N
50N
66N
88N
80 µm
20 µm
Ft (N)
AE (%)
Depth (µm)
Tangential Force (N), AE (%)
Depth (µm)
Applied Load (N)
0
20
40
60
80
100
120
0
-20
-40
-60
-80
-100
0
50
100
150
200

Adhesion on metals: Standard Rockwell (Daimler or Mercedes) tests

DLC on HSS

Cr + WC + DLC on HSS





Indentation

DLC delaminated coating

HSS substrate surface

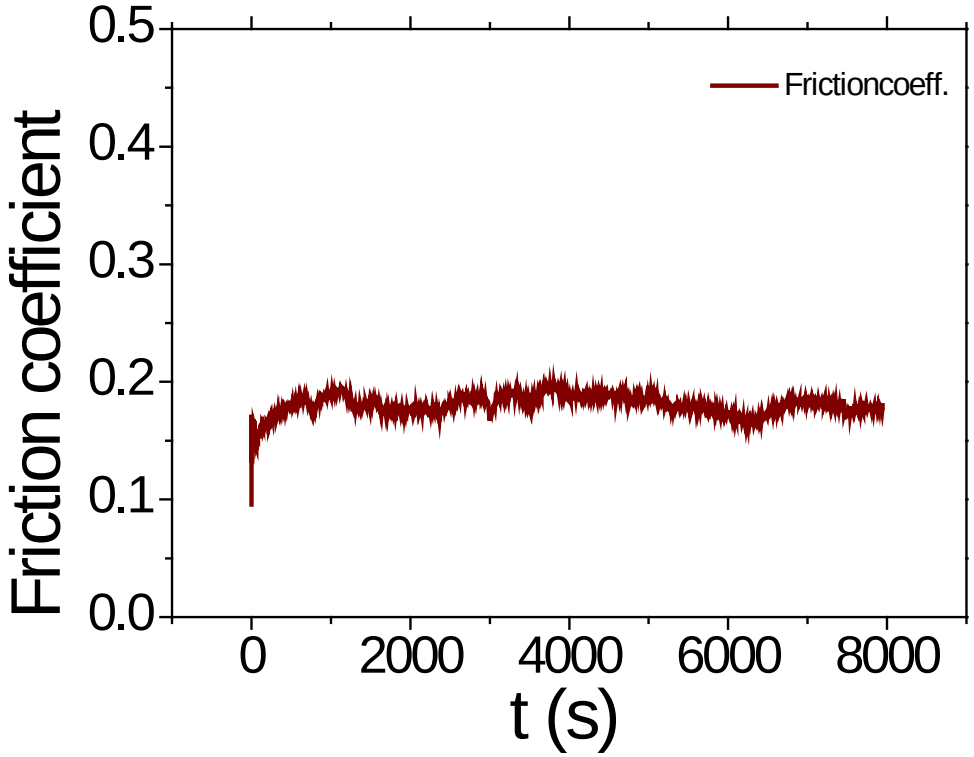
DLC coated surface

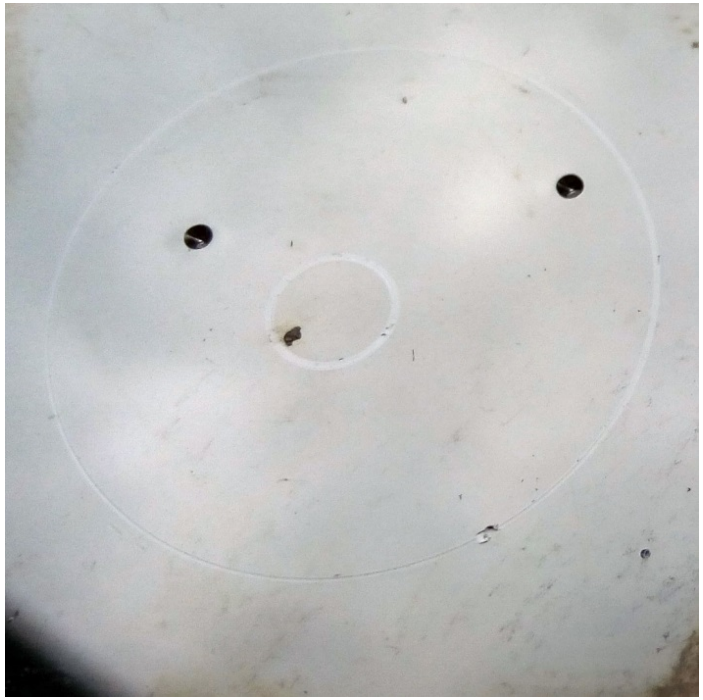
NO DLC coating delamination

Rockwell HRC tests - 150kg - Diamond indenter conical tip

Friction and wear – ASTMG99 Standard test

Friction and wear – ASTMG99 Standard test

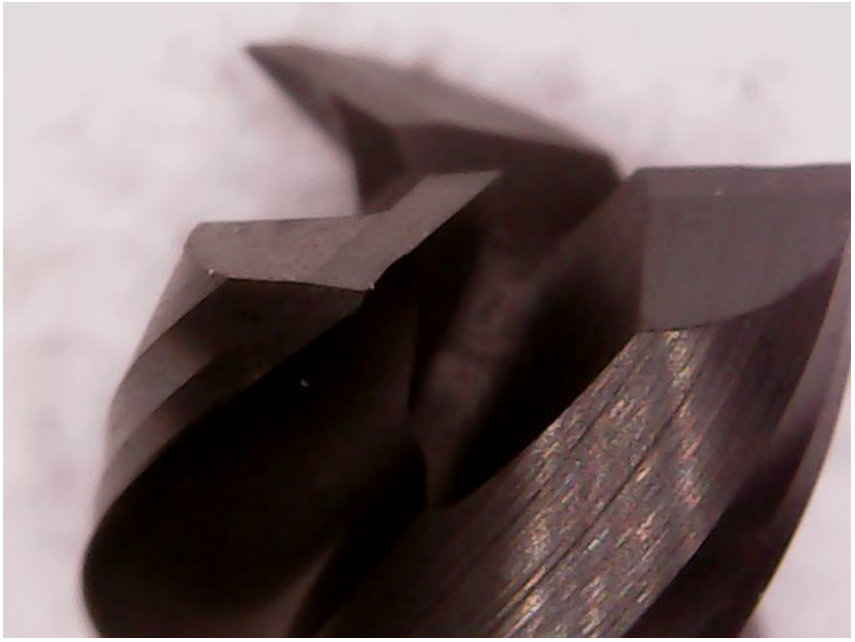




Low accumulated stress @ high hardness

Biased process : high DLC coating stress

N4E process : low DLC coating stress

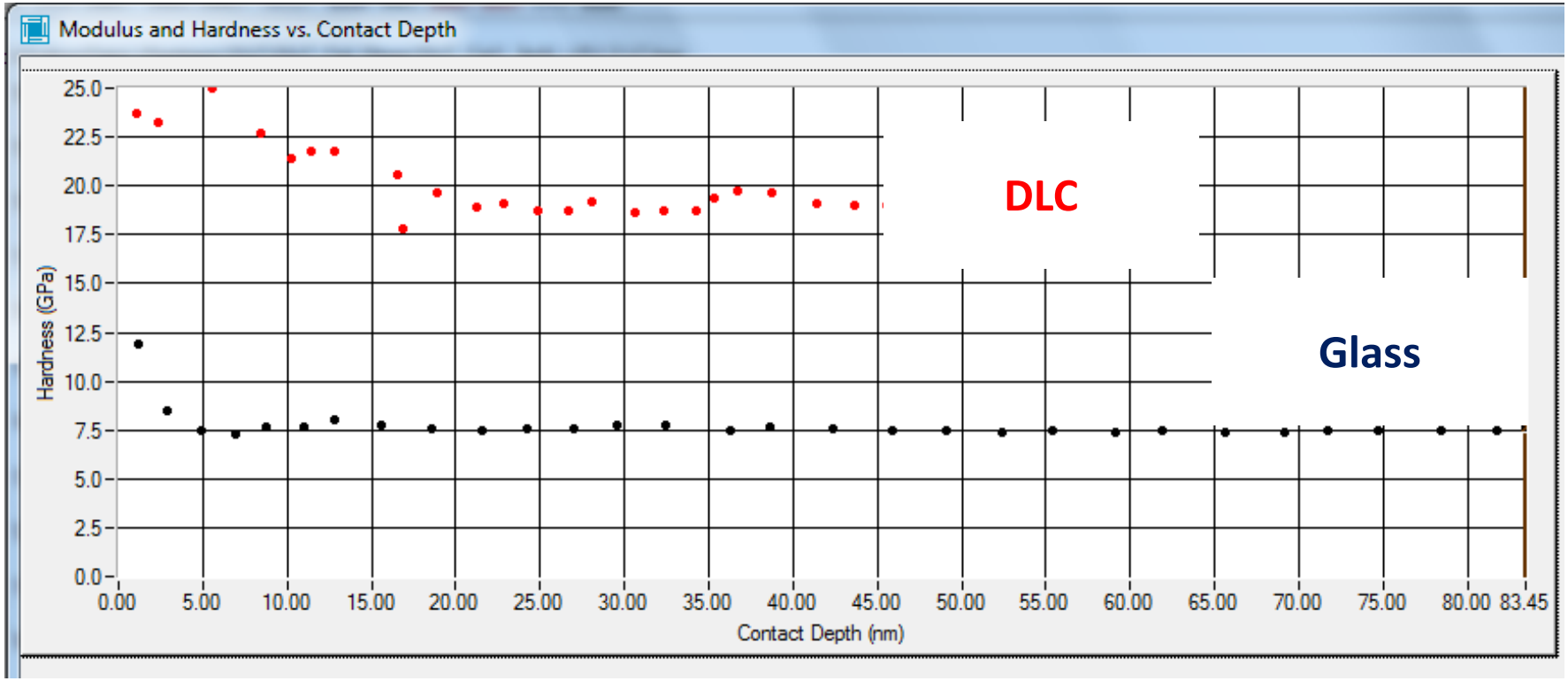


Ongoing stress measurements in DLC coatings

Insulating substrates : High hardness DLC for abrasion resistance

DLC coating on Glass

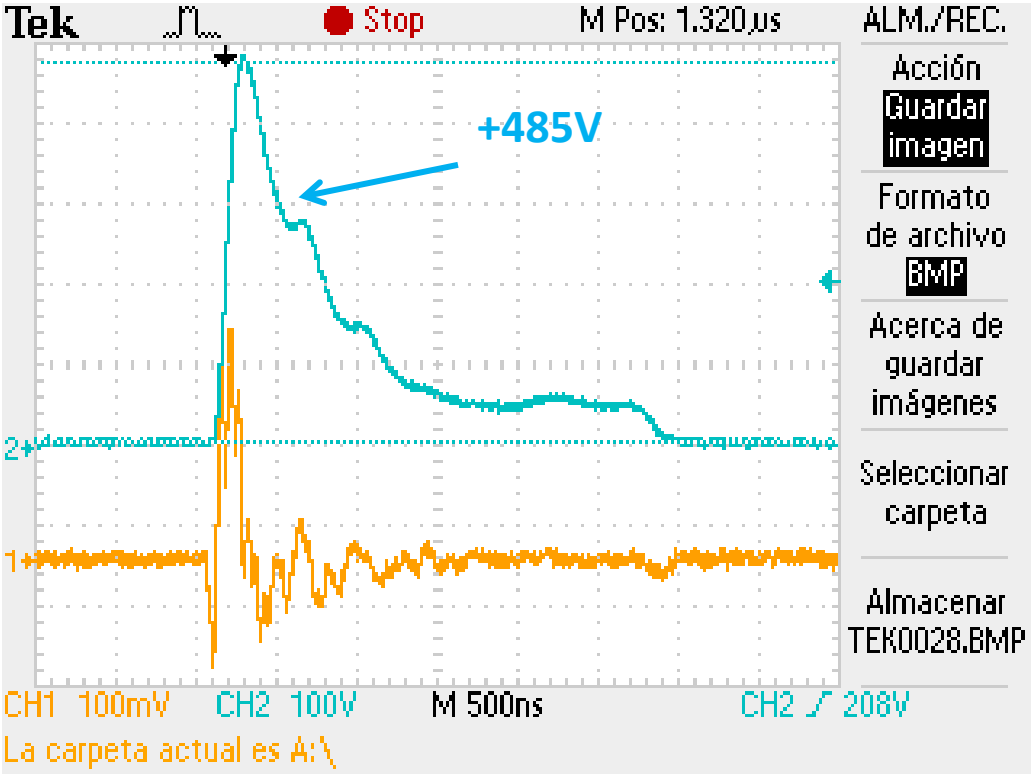
Delamination problem: Need to reduced thicknesses and ion bombardment



Hardness in the range of 20GPa for 40nm thick DLC layer

DLC coating on Glass

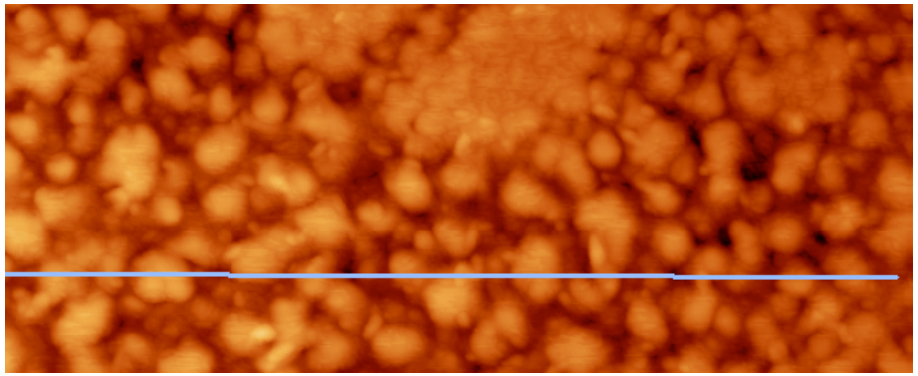
Substrate can be pre-treated with reactive gases such as oxygen



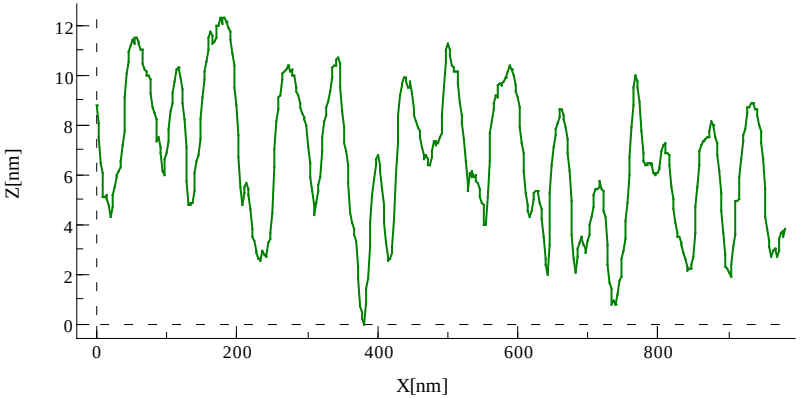
Current to floating potential (10mV/A)

DLC coating on Glass : substrate pre-treatment

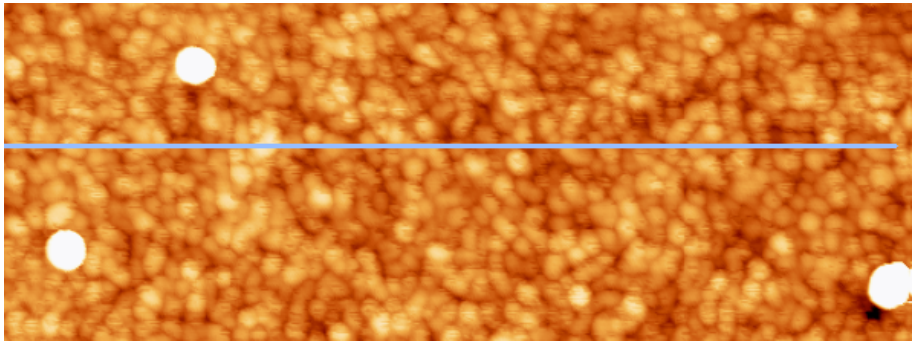
Untreated glass substrate



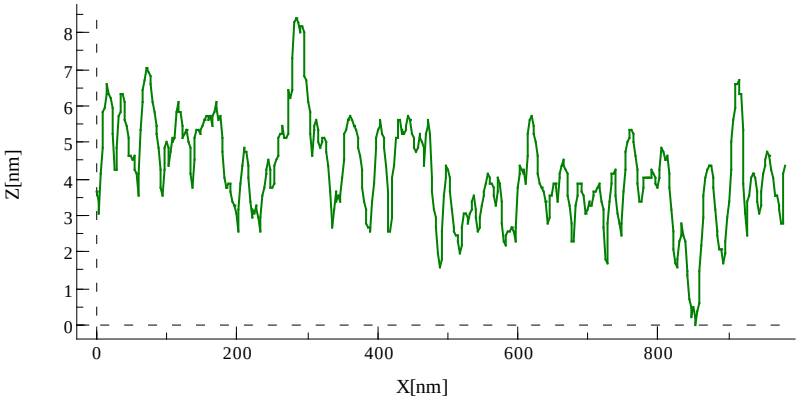
RMS roughness : 2.6 nm



Oxygen treated glass substrate



RMS roughness : 1.4 nm



DLC coating on Glass: Taber abrasion tests

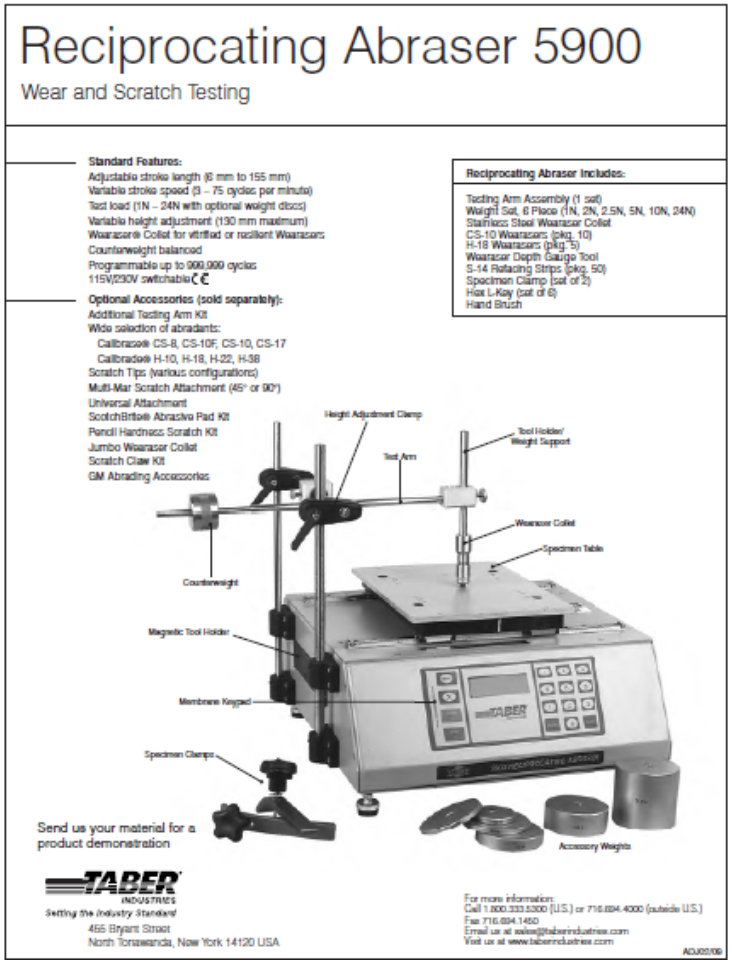
Coating description	TABER Test （7.5N）
DLC 1 : 30 min O2 plasma cleaning + 6nm DLC-a	≤20 times NG
DLC 3 : 15 min O2 plasma cleaning + 6nm DLC –a	≤20 times NG
DLC 2 : 30 min O2 plasma cleaning + 3nm DLC –a	≤20 times NG
DLC 6 : 5 min O2 plasma cleaning + 3nm DLC –a	**≤10 times NG**
DLC 4 : 15 min O2 plasma cleaning + 6nm DLC-b	≤20 times NG
DLC 7 : 5 min O2 plasma cleaning + 6nm DLC-b	**≤15 times NG**

Reciprocating Abraser 5900

Wear and Scratch Testing

TABER INDUSTRIES

ISO 9001:2000 CERTIFIED



Reciprocating Abraser 5900

Wear and Scratch Testing

Standard Features:
Adjustable stroke length (6 mm to 155 mm)
Variable stroke speed (3 – 75 cycles per minute)
Test load (1N – 24N with optional weight discs)
Variable height adjustment (130 mm maximum)
Wearaser® Collet for vitrified or resilient Wearasers
Counterweight balanced
Programmable up to 999,999 cycles
115V/230V switchable CE

Optional Accessories (sold separately):
Additional Testing Arm Kit
Wide selection of abradants:
Calibrase® CS-8, CS-10F, CS-10, CS-17
Calibrade® H-10, H-18, H-22, H-38
Scratch Tips (various configurations)
Multi-Mar Scratch Attachment (45° or 90°)
Universal Attachment
ScotchBrite® Abrasive Pad Kit
Pencil Hardness Scratch Kit
Jumbo Wearaser Collet
Scratch Claw Kit
GM Abrading Accessories

Reciprocating Abraser Includes:
Testing Arm Assembly (1 set)
Weight Set, 6 Pieces (1N, 2N, 2.5N, 5N, 10N, 24N)
Stainless Steel Wearaser Collet
CS-10 Wearasers (pkg. 10)
H-18 Wearasers (pkg. 5)
Wearaser Depth Gauge Tool
S-14 Refacing Strips (pkg. 50)
Specimen Clamp (set of 2)
Hex L-Key (set of 6)
Hand Brush

Send us your material for a product demonstration

TABER INDUSTRIES
Setting the Industry Standard
455 Bryant Street
North Tonawanda, New York 14120 USA

For more information:
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Fax 716.694.1450
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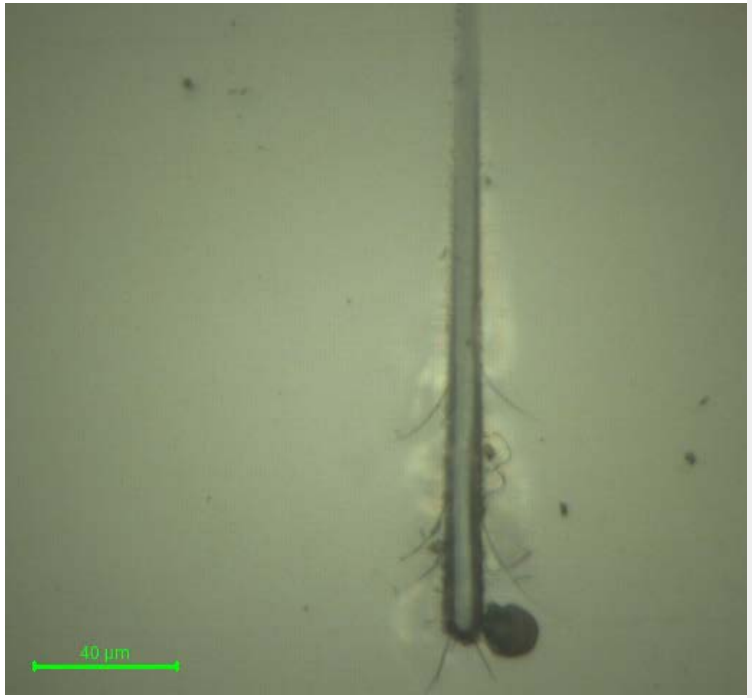
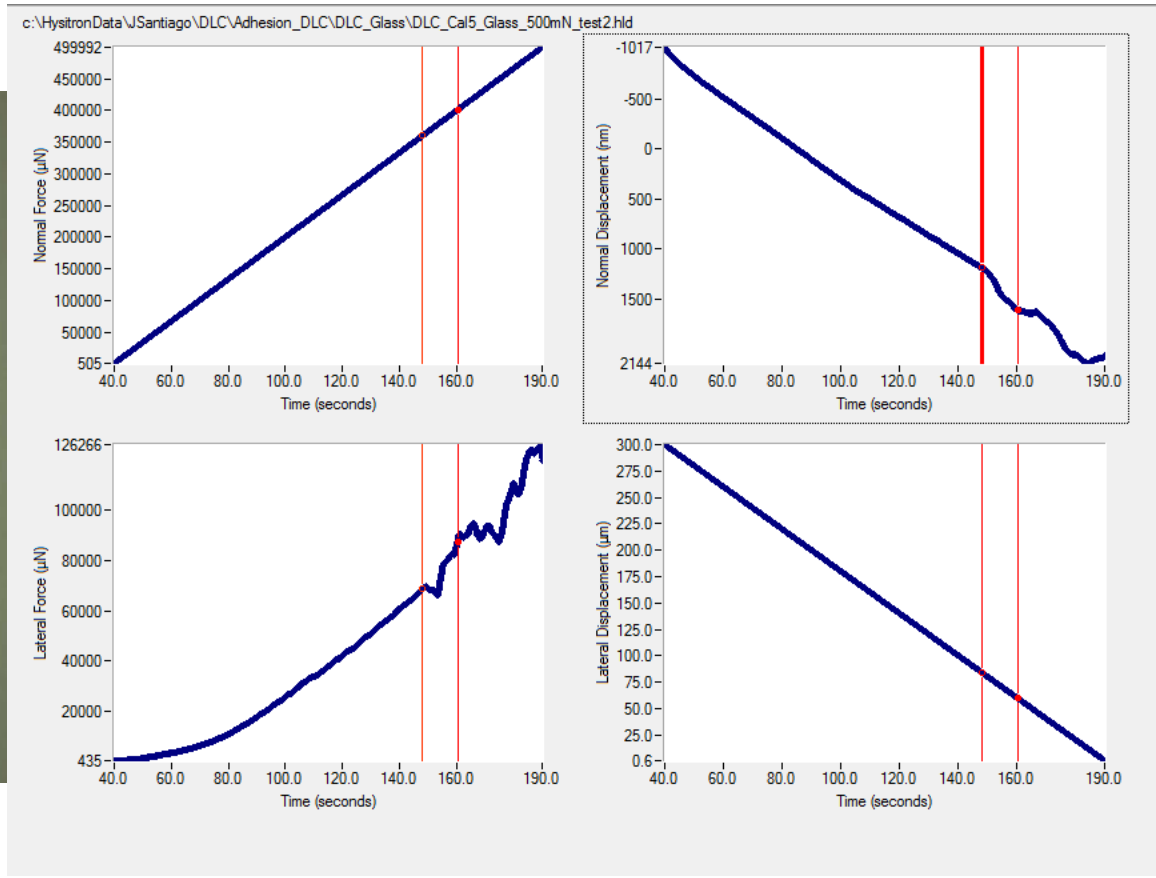
Visit us at www.taberindustries.com

ADJ02/09

Substrate plasma pre-treatment improved DLC coating adhesion

DLC coating on Glass: nano-scratch tests





500mN critical load – 5 micron tip radius

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Thank you for your attention!