Characterization of DLC Coatings Deposited by Large Area Filtered Arc Deposition (LAFAD^{TM}) Technique

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Discussion Topics

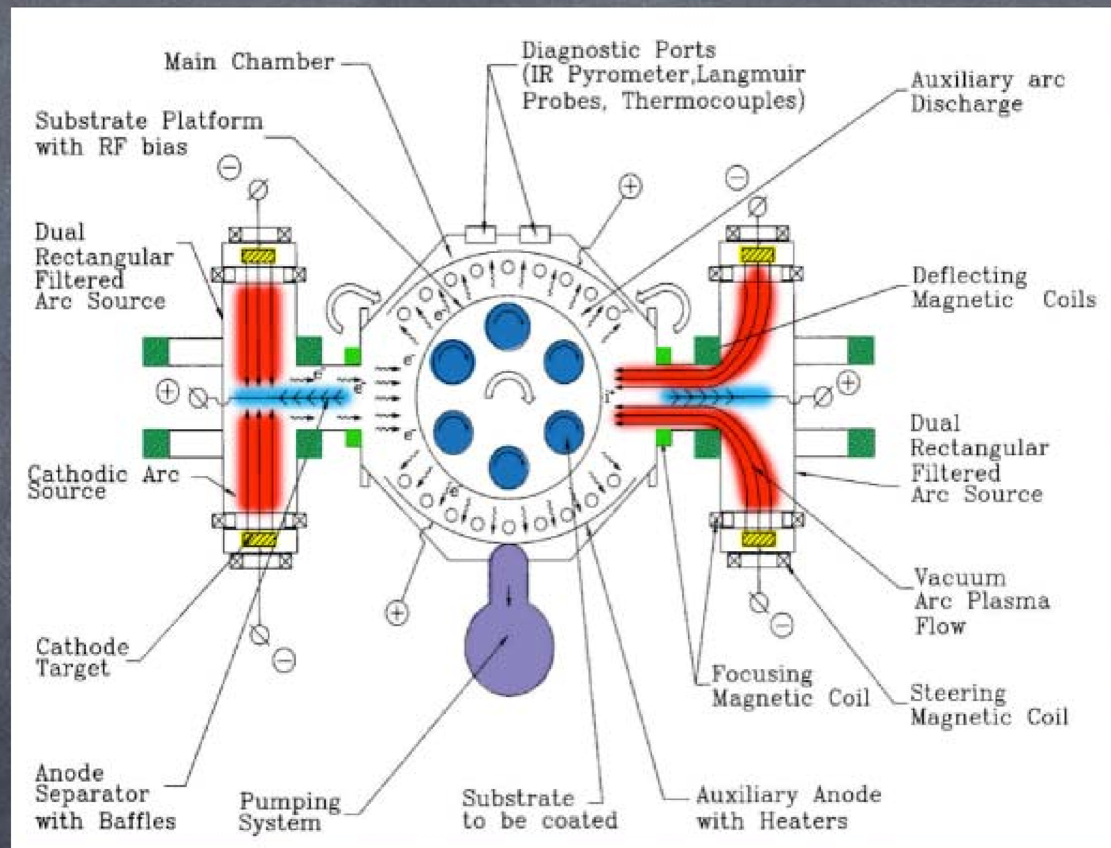
- **Motivation for Research**
  - **Understanding the role of processing parameters on mechanical properties and coating/substrate interface of H-free DLC on different substrates**
- **Technical Approach**
  - **Large Area Filtered Arc Deposition (LAFAD^{TM}) technology for deposition of H-free DLC**
  - **EDS, RBS and cross-sectional analysis for investigating the coating/substrate interface**
  - **Various techniques for investigating mechanical properties: scratch test, nano-indentation, wear-scar CALOtest**
- **Basic coating characteristics**
  - **Thickness, adhesion, composition, elemental distribution across coating/substrate interface**
- **Objective: highlight LAFAD^{TM} technology for deposition of hydrogen free DLC coatings**

Large Area Filtered Arc Deposition
LAFAD

In Situ Process & Equipment Drawing featuring multiple cathodes, multiple material element combination

- Cathode Arc Sources
- Deflecting Magnetic Coils
- Steering Magnetic Coil
- Plasma Stream
- Anode Separator (Filter)
- Substrate Holders
- Vacuum Pump

In Situ Plasma Stream



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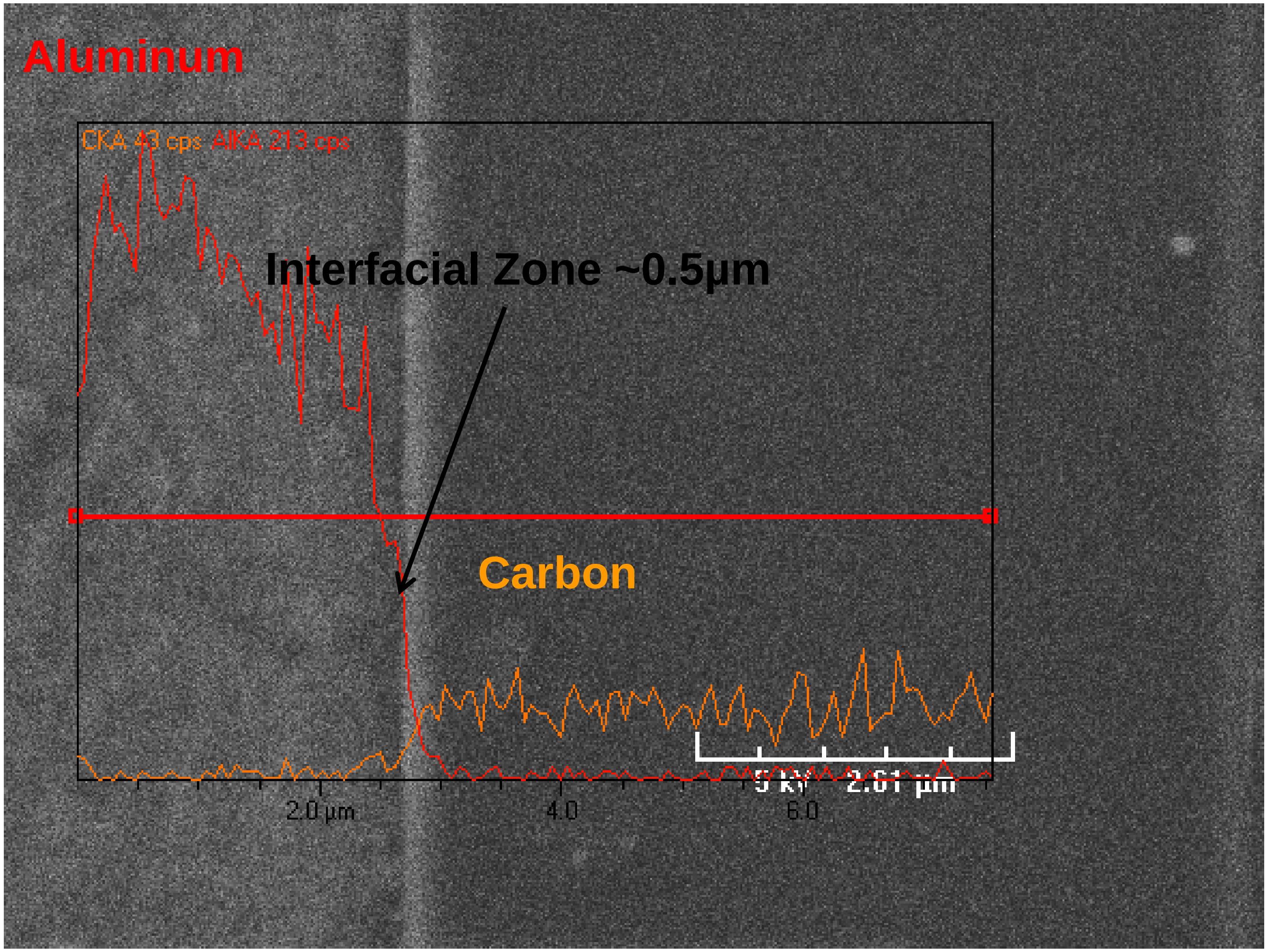
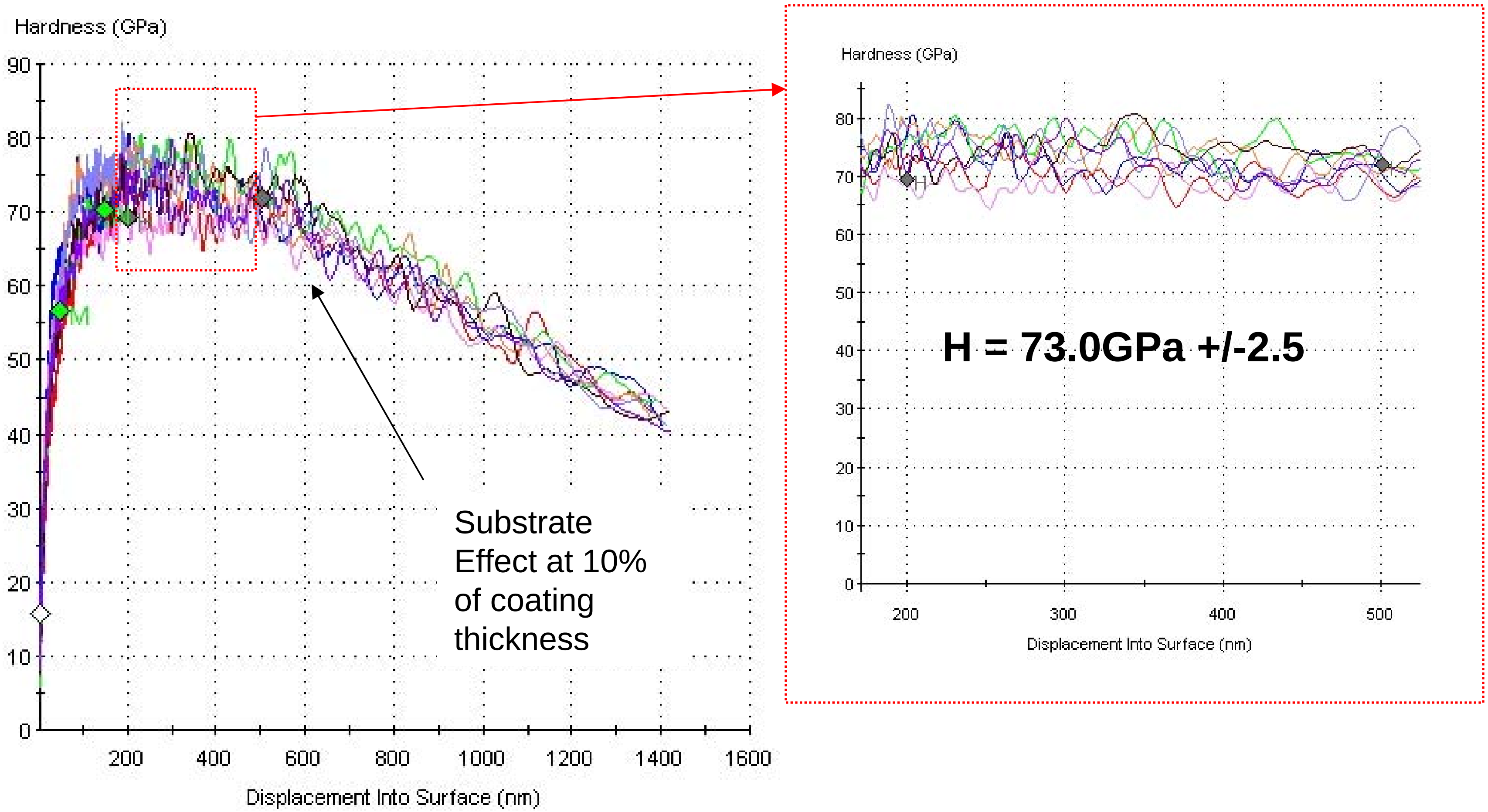
Photograph of graphite cathodic arc target used for deposition of DLC coatings

Elemental distribution by SEM/EDS technique across relatively thick (~6um) DLC film on aluminum. The film was deposited by LAFAD[TM] source with graphite targets on water-cooled aluminum disk under RF bias with autopolarization potential ~100 volts (estimated C+ ion bombardment energy during coating deposition ~100eV).



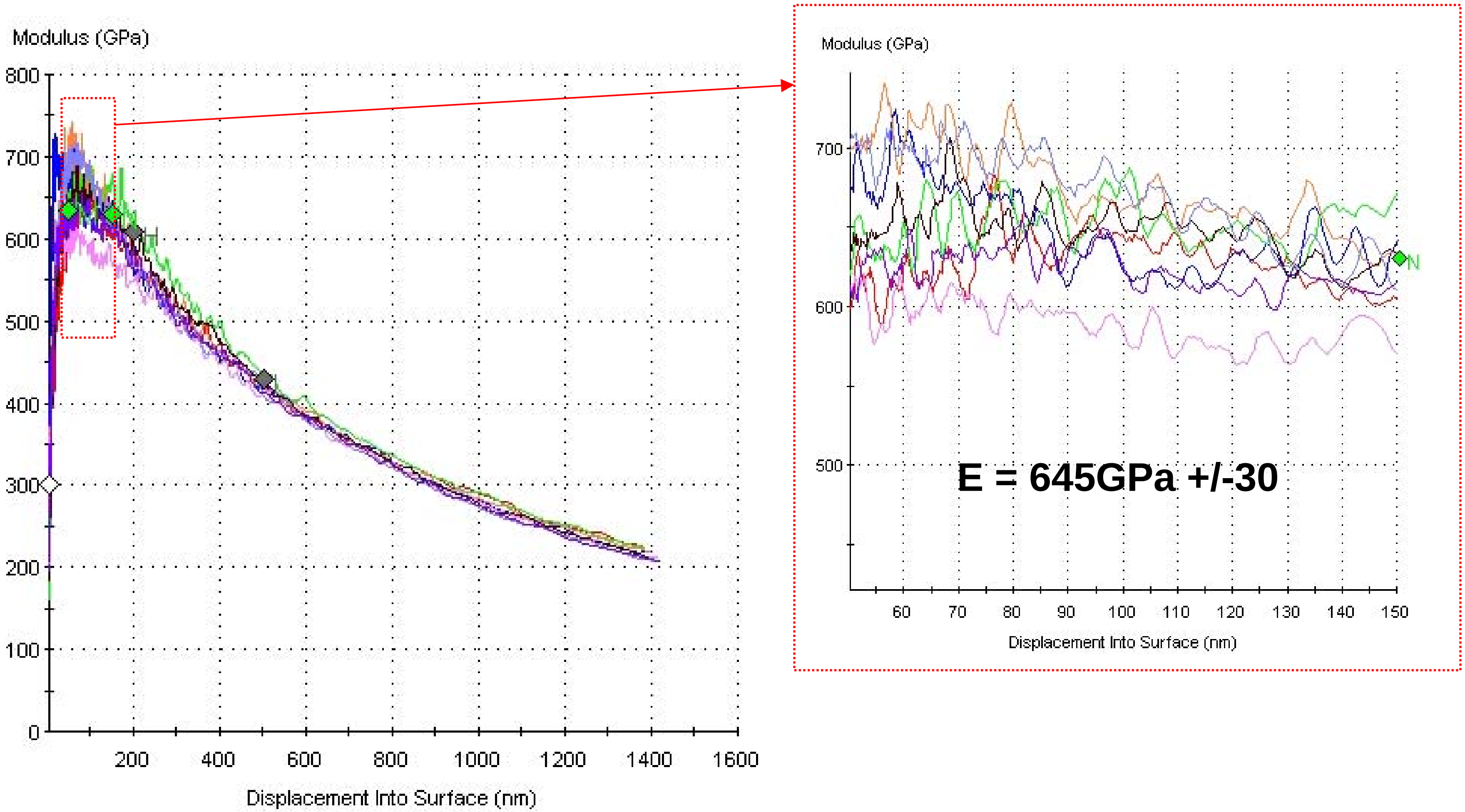
Hardness (H) of DLC (6um) on Al

- MTS Nanoindenter XP, Continuous Stiffness Module (CSM) Thin Film Method
- 8 indents on coating, 1400nm total displacement, Berkovich tip
- Mean hardness calculated over 200-500nm displacement (~4-8% of coating thickness)



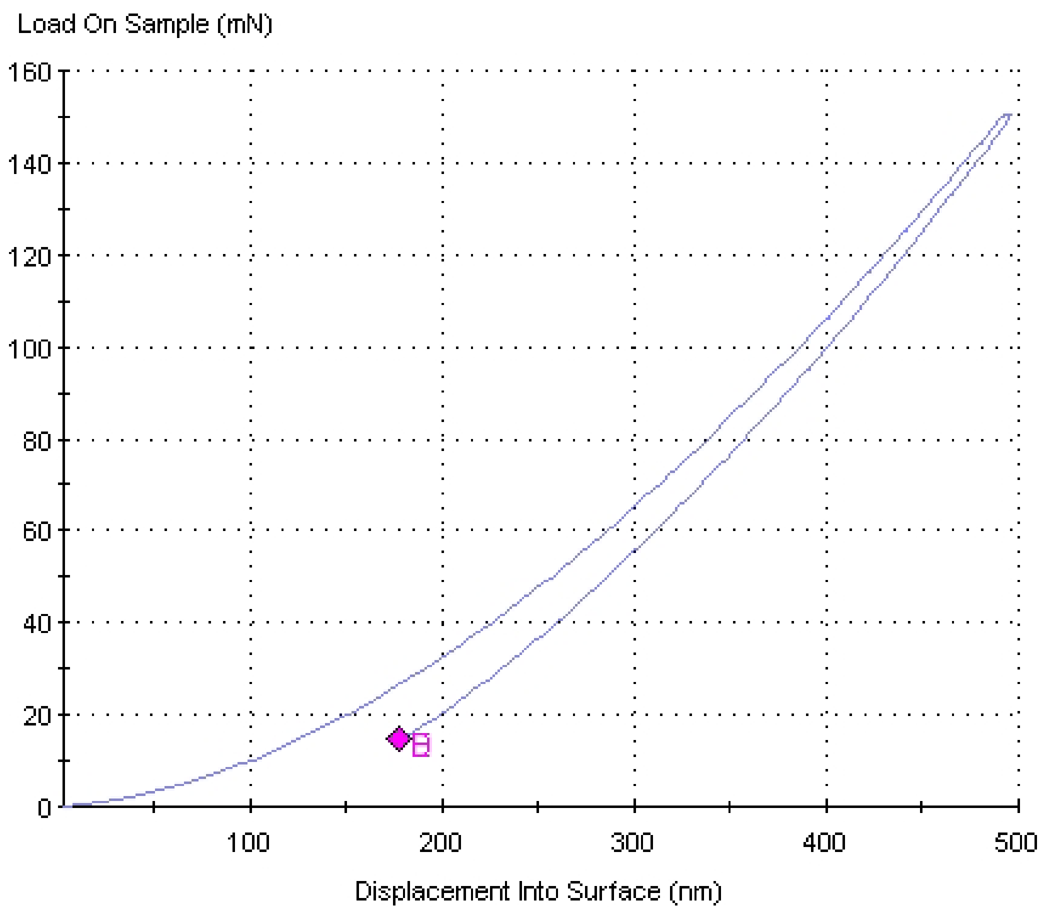
Elastic Modulus (E) of DLC (6um) on Al

- MTS Nanoindenter XP, Continuous Stiffness Module (CSM) Thin Film Method
- 8 indents on coating, 1400nm total displacement, Berkovich tip
- Mean modulus calculated over 50-150nm displacement (see figures)



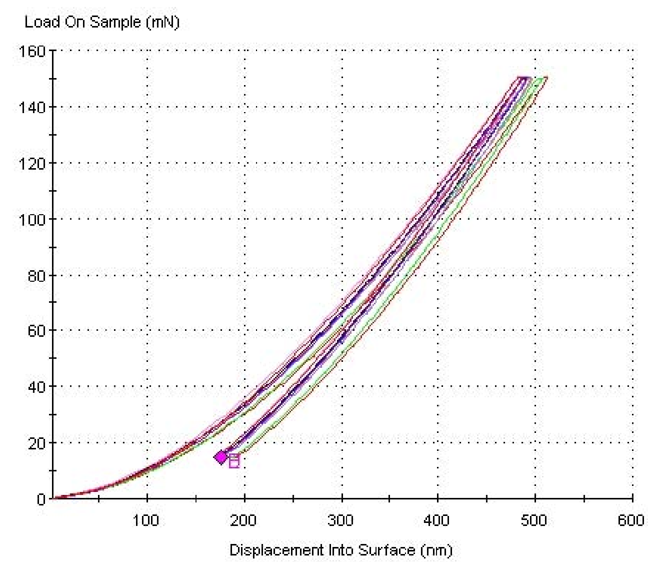
Load Displacement Curve for DLC (6um) on Al

- MTS Nanoindenter XP, Load Control Method (traditional single load and unload)
- 9 indents on coating, 15.3mg (150mN) load, Berkovich tip
- ~4nm of drift during 20sec max load hold time (should not be related to thermal drift)



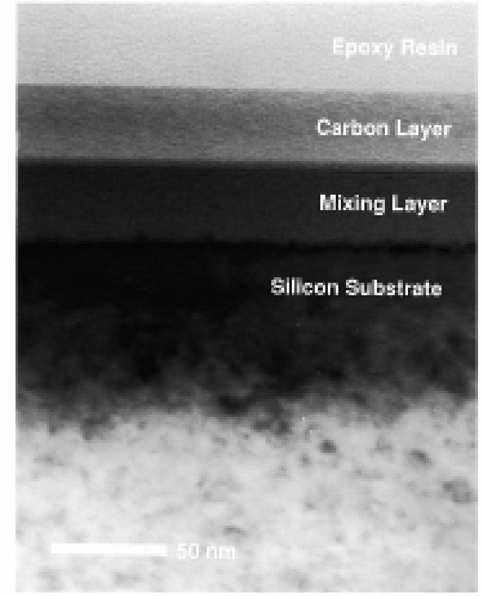
H = 71.2GPa +/-4.4

E = 423GPa* +/-17

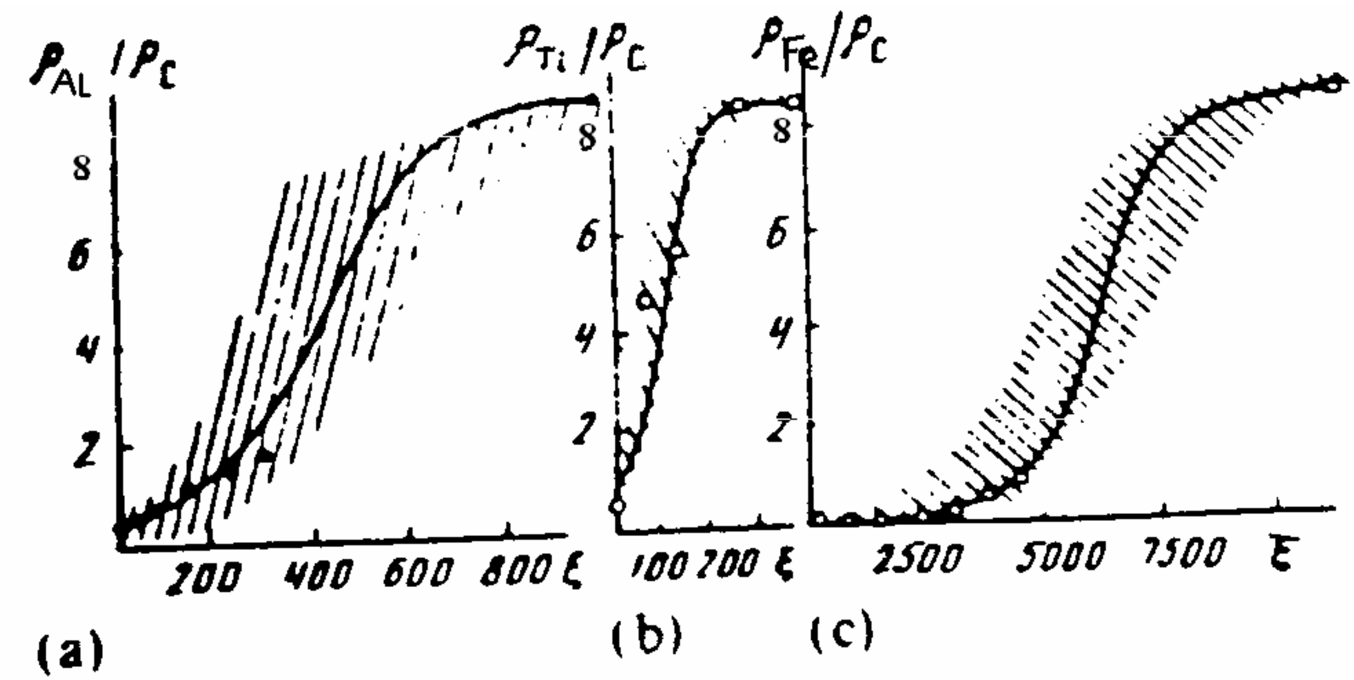


* Elastic modulus calculated from unload using Berkovich tip and 150mN load may not be accurate (spherical indenter should be used, to validate modulus measurement)

Examples of interfacial layers forming by hydrogen free DLC deposition on different metal substrates with ion bombardment energy of C^+ ions of ~100 eV. These films were deposited by filtered arc process on water cooled substrates at 10^{-3}Pa residual gas pressure. The RF 1,76 MHz bias provided 50-100 volts autopolarization potential between substrate and carbon plasma.



Forming interfacial layer between DLC and Si. Ion bombardment energy of C+ ions in this work was ~100 eV. After D.R.McKenzie, 1994



The ratio of substrate-to-carbon atoms in DLC/substrate interfacial layer: a- DLC/Al; b- DLC/Ti; c- DLC/Fe. Note: the depth ξ is shown in distance units between atomic layers, h. The points are experimental results obtained by Auger with 3kV H+ beam etching. The cross-hatched area represents the variations in calculation results due to different input parameters (after N.V.Novikov et al., 1990).

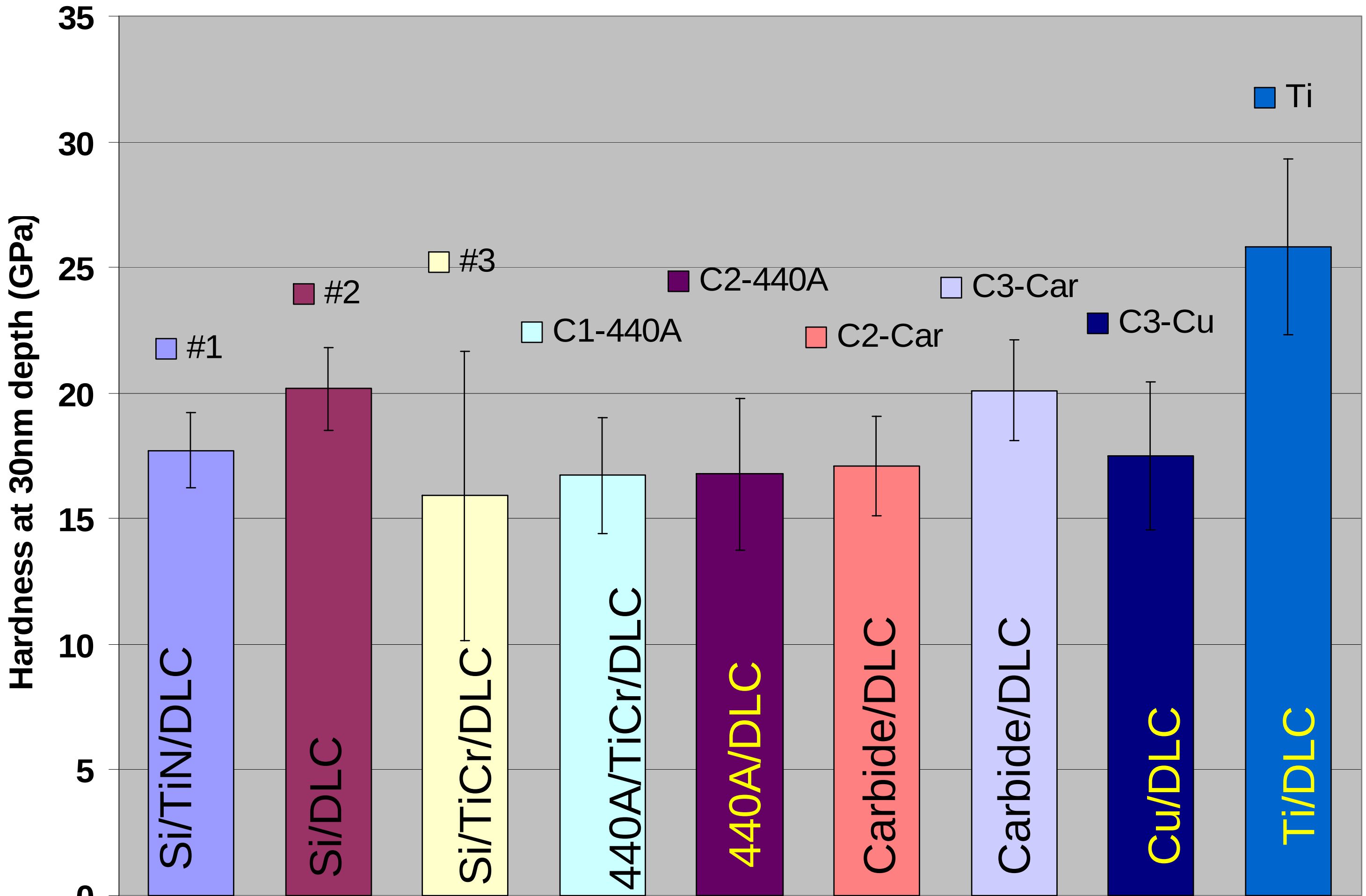
Process settings for deposition of thin DLC films (thickness <0.5um) by Large Area Filtered Arc Deposition (LAFAD^{TM}) technique

The rest of the coating samples investigated in this work were prepared by dual arc LAFAD^{TM} process with the following processing conditions:

- **Total residual gas pressure 10^{-3}-10^{-2} Pa (influenced by outgasing of the graphite cathode target)**
- **Floating bias with HV pulse assistance**
- **HV pulse amplitude 2.5 kV, pulse width 25 us, repetition frequency 600 Hz**
- **Average C^{+} ion impact energy ~30eV.**

Arcomac LAFAD DLC coatings

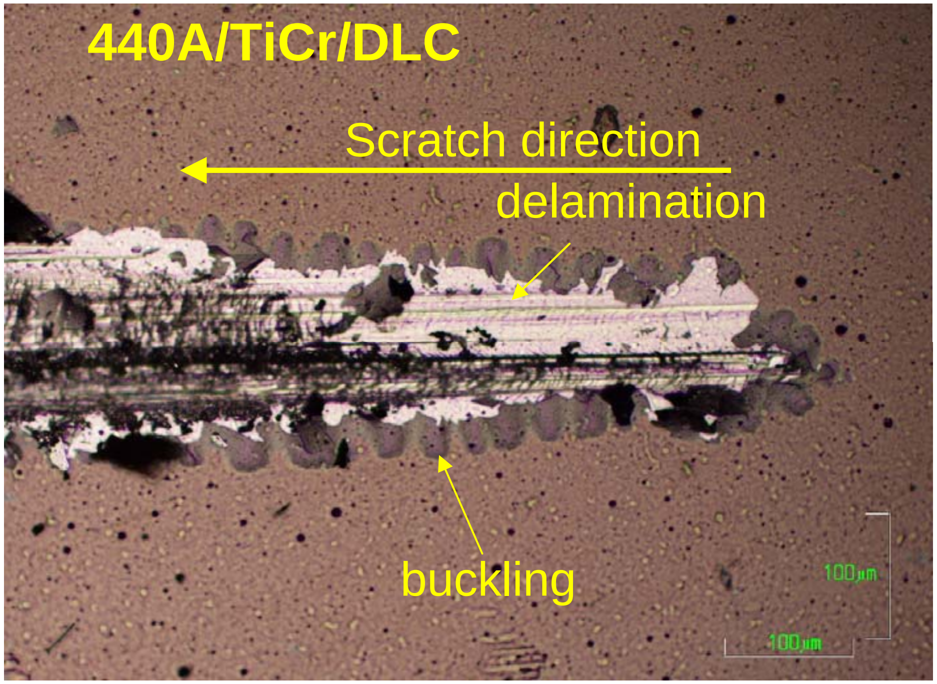
- Average over 10 nanohardness measurements per sample at depth of 30nm
- Hardness ranged from 16-20 GPa (25.8 for Ti/DLC)

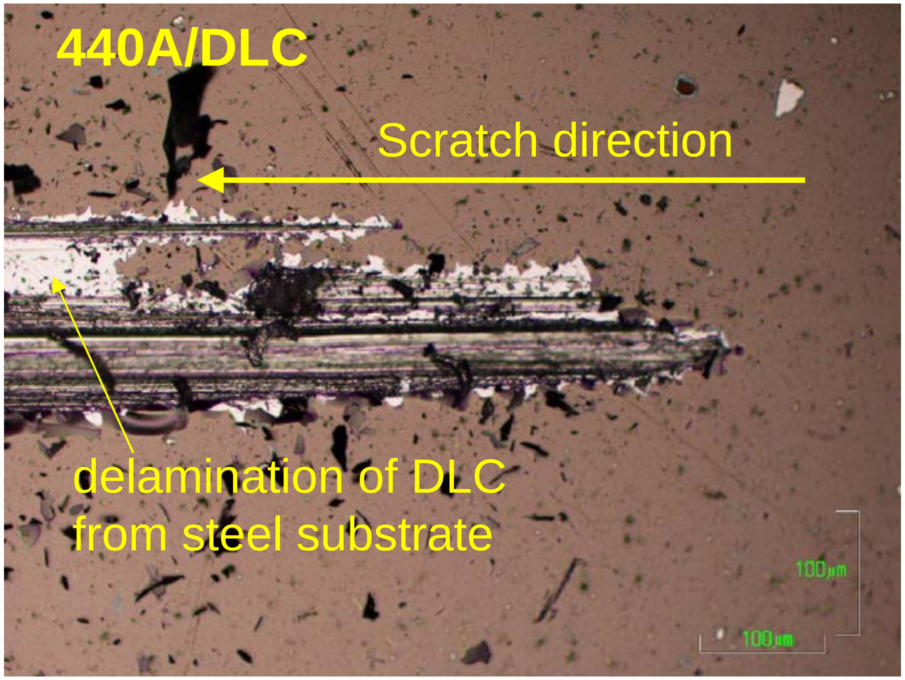


Arcomac DLC coatings – scratch testing

• 440A/TiCr/DLC: Peripheral buckling observed but no delamination beyond scribe

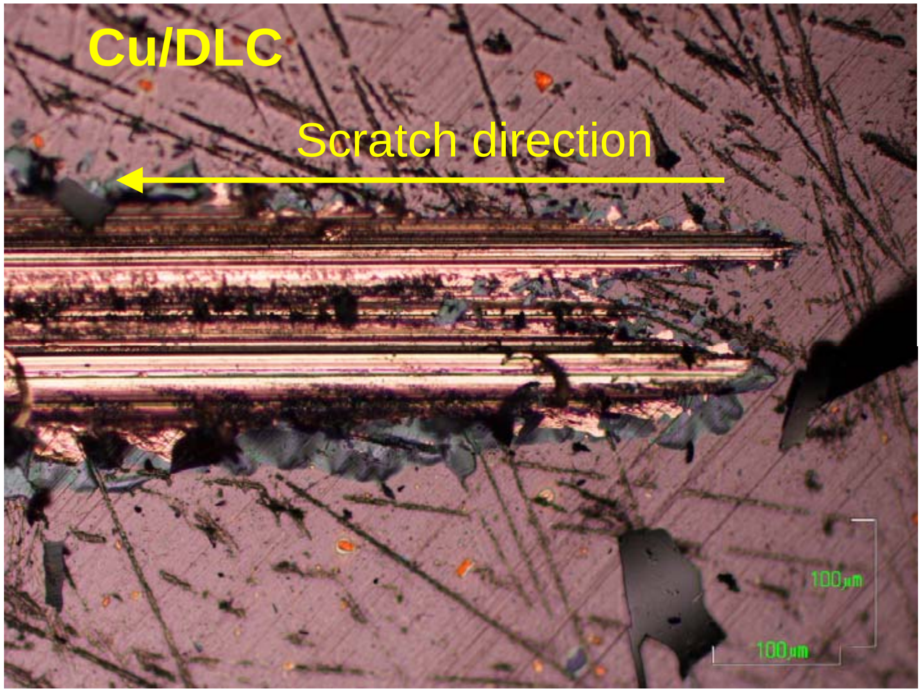
• 440A/DLC: Peripheral delamination of DLC from steel substrate occurred

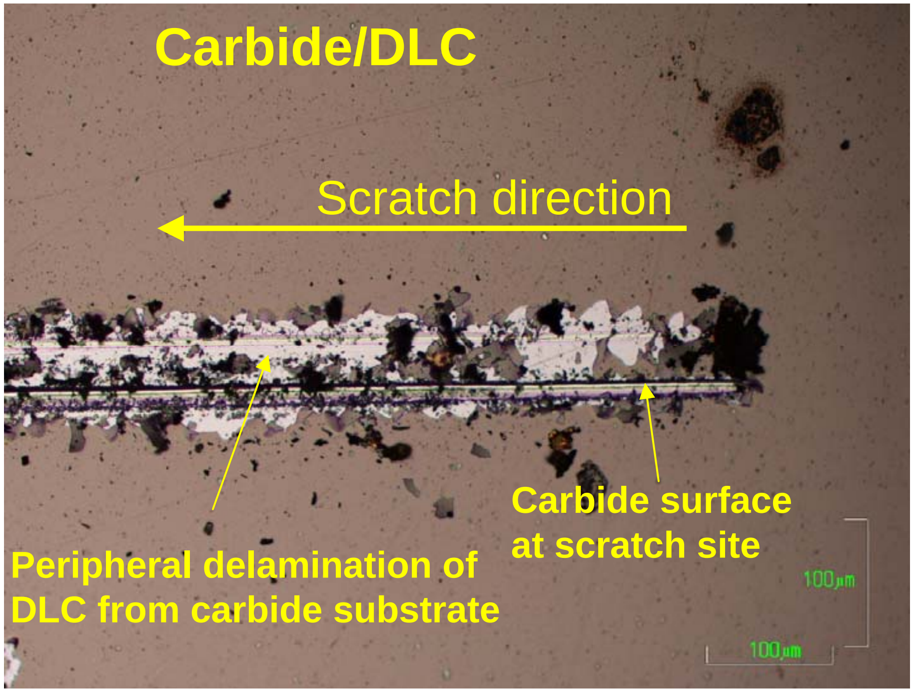




Arcomac DLC coatings – scratch testing

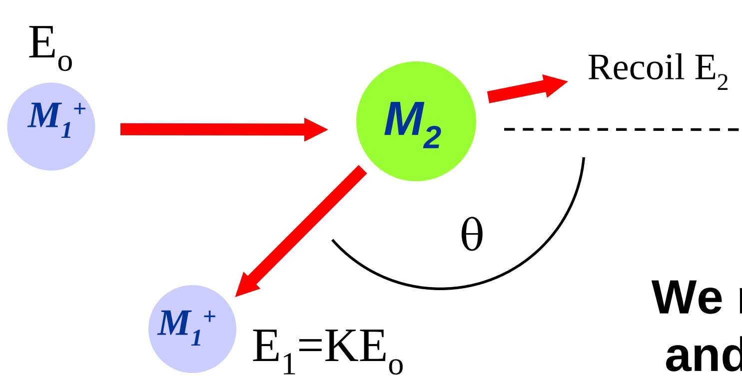
• Cu/DLC: Good adhesion between DLC and Cu substrate – no peripheral delamination observed
• Carbide/DLC: Peripheral delamination of DLC from carbide substrate was observed





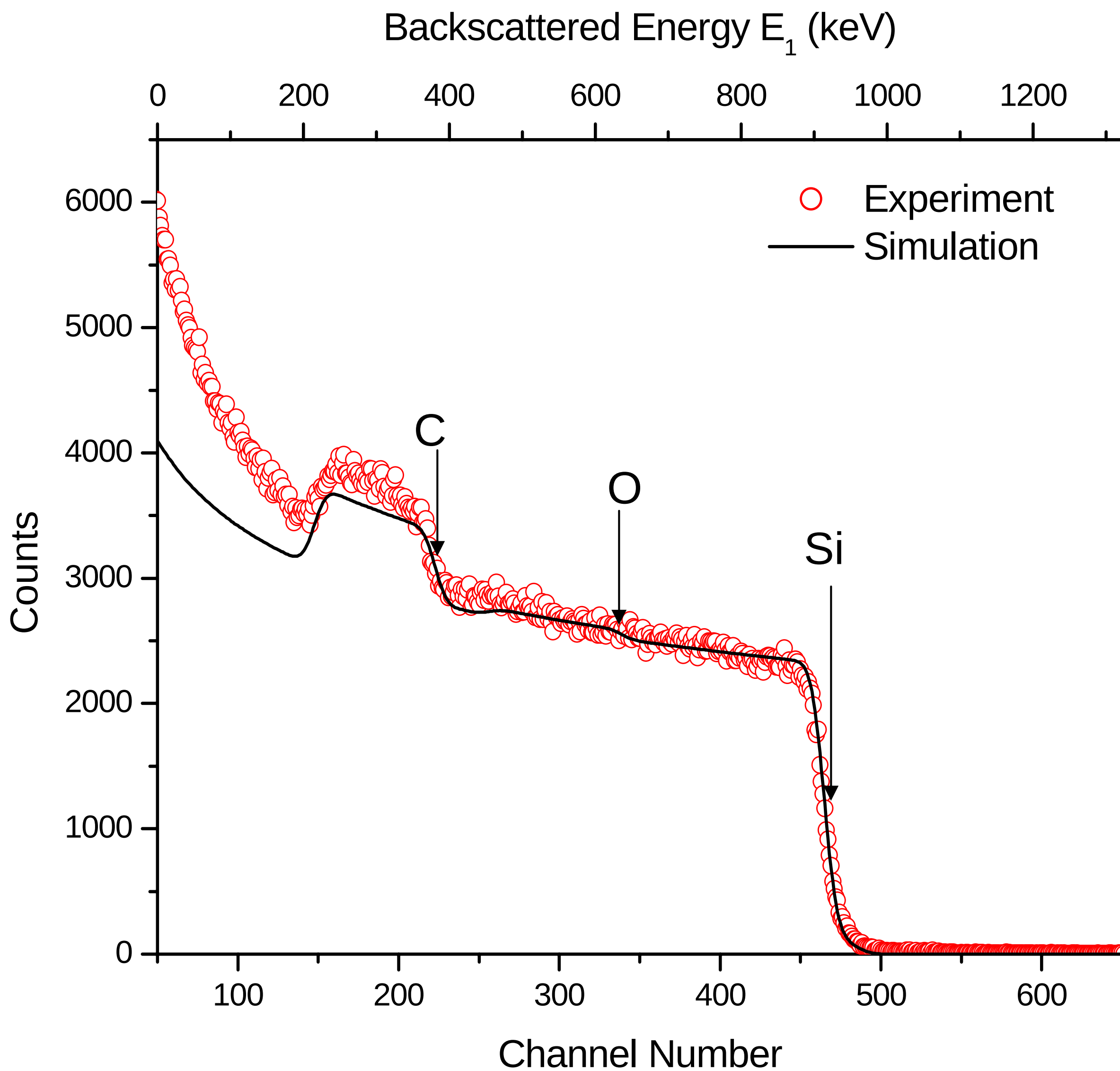
Rutherford Backscattering Spectrometry (RBS)

- Incoming ions are scattered from atoms inside the film
- The energy of the backscattered ion depends on the mass of the target atom and scattering angle through the kinematic factor, K



$$K = \left[\frac{\left(M_2^2 - M_1^2 \sin^2\theta\right)^{1/2} + M_1 \cos\theta}{M_1 + M_2} \right]^2$$

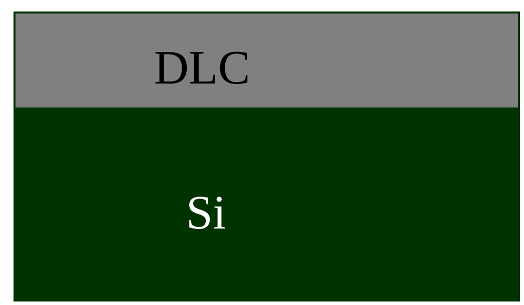
We measure E1 (backscattered ion energy) and that includes 2 contributions:
One is energy loss of the collision and second is energy loss in traveling throughout the film.
From the factor K we can calculate the mass of The target atoms.

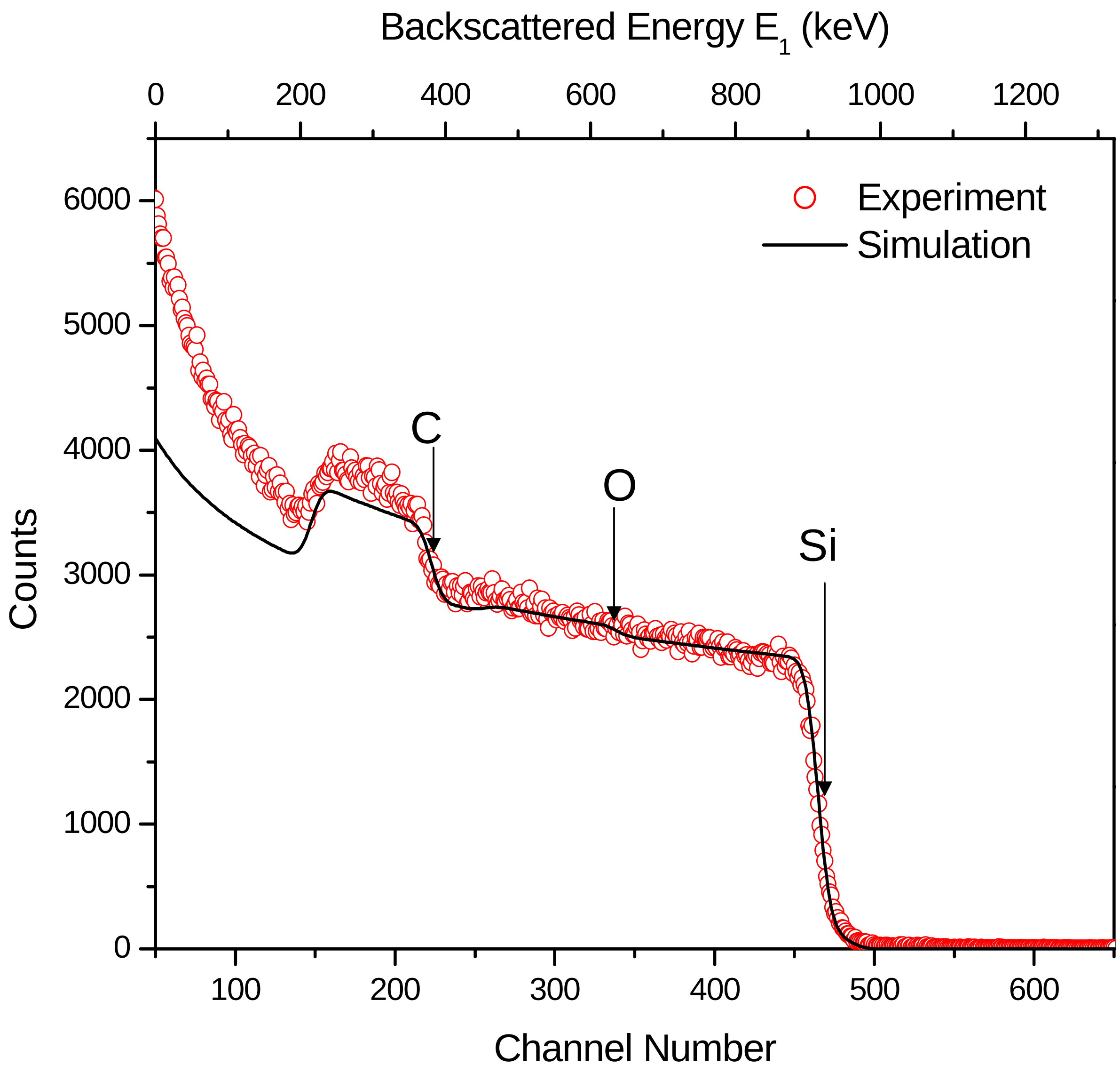


Sample C/Si (#3-87-1)

RBS, 2 MeV He^+ beam

Layer #	Elements	Thickness (10^{15}Atoms/ cm^2)
1	94% C, 6% O	3026
2	100% Si	substrate





Sample C/Cu (3-87-1)

NRBS, 1.23 MeV H^+ beam

Layer #	Elements	Thickness (10^{15} Atoms/ cm^2)
1	86% C, 4% N, 10% O	2950 (~0.21µm)
2 Subst -rate	100% Cu	

C/Cu (3-87-1)

Layer #	Elements	Thickness (10^{15}Atoms/cm^2)
1	86% C, 4% N, 10% O	2950 (~0.21µm)
2	100% Cu	substrate

DLC/Ti6Al4V (12-28-06)

Layer #	Elements	Thickness (10^{15} Atoms/cm^2)
1	95% C, 5% O	1910 (~0.14µm)
2	45% Ti, 4% Al, 8% O, 43% C	600 (Å)
3	90% Ti, 6% Al, 4% V	substrate

C/Al (12-28-6)

Layer #	Elements	Thickness (10^{15}Atoms/cm^2)
1	81% C, 19% O	2270 (~0.16µm)
2	83% Al, 10% O, 7% C	2500 (Å)
3	90% Al, 10% O	1300
4	100% Al	substrate

No DLC/Substrate interface needed to fit Cu substrate data, but do see C/Ti6Al4V interface and thicker C/Al interface

DLC/Si (3-87-1)

Layer #	Elements	Thickness (10^{15}Atoms/cm^2)
1	94% C, 6% O	3026 (~0.22μm)
2	100% Si	substrate

DLC/TiON/Si (12-28-6)

Layer #	Elements	Thickness (10^{15}Atoms/cm^2)
1	94% C, 6% O	2570 (~0.18μm)
2	41% Ti, 27% N, 31% O,	955 (Å)
3	25% Ti, 30% N, 25% O, 20% Si	289
4	100% Si	substrate

DLC/TiCr/Si (12-28-6)

Layer #	Elements	Thickness (10^{15}Atoms/cm^2)
1	94% C, 6% O	2390 (~0.17μm)
2	28% Cr, 51% Ti, 21% O	>9500 (Å)
3	100% Si	substrate

No DLC/Substrate interface needed to fit data but do see Si/TiON interface

DLC/440A (12-28-6)

Layer #	Elements	Thickness (10^{15}Atoms/ cm^2)
1	90% C, 6% O, 4% N	2700 (~0.19µm)
2	82% Fe, 18% Cr	substrate

DLC/CrTi/440A (12-28-6)

Layer #	Elements	Thickness (10^{15}Atoms/ cm^2) Thickness (10^{15}Atoms/ cm^2)
1	90% C, 6% O, 4% N	2810 (~0.20µm)
2	65% Ti, 35% Cr	14600 (Å)
3	82% Fe, 18% Cr	substrate

DLC/440C BB (ball bearing) (3-87-1#4)

Layer #	Elements	Thickness (10^{15}Atoms/ cm^2)
1	92% C, 3% O, 5% N	2640 (~0.19µm)
2	22% Fe, 39% Cr, 30% C, 3% N, 5% O	775 (Å)
3	43% Fe, 30% Cr, 18% C, 4% N, 4% O	589 (Å)
4	60% Fe, 28% Cr, 4% C, 4% N, 4% O	887 (Å)
5	66% Fe, 22% Cr, 2% C, 5% N, 5% O	1790 (Å)
6	71% Fe, 19% Cr, 0.4% C, 4% N, 5% O	1600 (Å)
7	82% Fe, 18% Cr	substrate

No DLC/substrate interface needed to fit 440A with and without CrTi interlayer

RBS data suggest interdiffusion at DLC/440C interface (ball bearing).

Summary

- **Large Area Filtered Arc Deposition (LAFAD™) technology was used for deposition of hydrogen-free carbon DLC coating on substrates made of different metals.**
- **Coating composition and elemental distribution across the films was studied by RBS and EDS technique; computer modeling was used to estimate the composition and thickness of the interfacial layers in the coatings.**
- **Mechanical properties were studied by nanoindentation and scratch test techniques**
- **It was found that width of interfacial layer between substrates and the DLC films deposited at low temperatures (<100C) depends upon bias potential and thickness of the film (process duration)**
- **In some cases the interfacial layer can be formed between DLC film and substrate even at floating bias and near ambient temperature conditions**
- **It was found that thin films (thickness ranging from 0.2 to 0.5um) deposited on different metal substrates under floating bias, show the hardness ranging from 15 to 25GPa.**
- **The hardness of relatively thick (~6um) DLC film deposited on water cooled Al by C+ ions with ~100eV bombardment energy generated by LAFAD™ source is ~73GPa**
- **Adhesion behavior varies depending on type of substrate with good adhesion on soft Cu substrate vs. brittle delamination on carbide**
- **Adhesion improvement on TiCr coated vs. uncoated 440A steel can be explained by influence of the TiCr layer between DLC film and the substrate**

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