# Plasma Focus assisted carburizing of aluminum at room temperature

Presented by
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With thanks to
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# Layout of the Talk

- Motivation
- Experimental Setup
- Diagnostics
- Results
- Conclusion

- The present work reports the selected results of carbon-ions implantation in aluminum specimens at the room temperature by using a Mather type plasma focus device, operated with methane (*a new process of plasma carburizing*), fired at 18kV (1.45kJ), giving a peak discharge current of about 160kA over multiple shots.

# Motivation

- Aluminum and its alloys are popular for their extensive use in automotive, rail, marine and aerospace industries.
- It is widely used in electrical shielding, electronic circuits and devices making, food and pharmaceutical packaging, water treatment and house decoration owing to its nonmagnetic, very low sparking, less trashing, nontoxic and noncombustible behavior.

- Its outstanding properties of light weight, good malleability and formability, high corrosion resistance, excellent electrical and thermal conductivity, better light reflectivity and good recyclability are making the aluminum an essential and ideal metal for today's advanced technological world.

- However, its insufficient wear resistance with low thermal and chemical stability limits its use for commercial and industrial purposes. The high strength to weight ratio of aluminum lures for its application in the area of sliding machine parts for weight saving purpose but its vulnerability to seizing and the low surface hardness (lower than that of steel and iron alloyed materials mainly used for these parts at present) offer a strong barrier.

- Therefore surface modification of aluminum, to improve wear and corrosion resistance, surface hardness and lower coefficient of friction, is intensively required to enhance its utilization in all fields of interest.
- Presently, nitriding, oxidizing and carburizing of aluminum by employing different techniques are commonly used to obtain tribologically and mechanically improved aluminium surface.

- Nitriding of aluminum is the most studied and investigated process for surface modification to achieve desired combination of physical and chemical properties. like high hardness, excellent wear resistance, high electrical and corrosion resistance, very good optical properties, and high thermal and chemical stability.

- In spite of all the properties the AlN exhibits, the surface of AlN layer is often morphologically rough containing deep grooves and cracks and is considered unsatisfactory for industrial application.
- Also the physical parameters' effects on tribological and mechanical properties of AlN are not yet well understood.

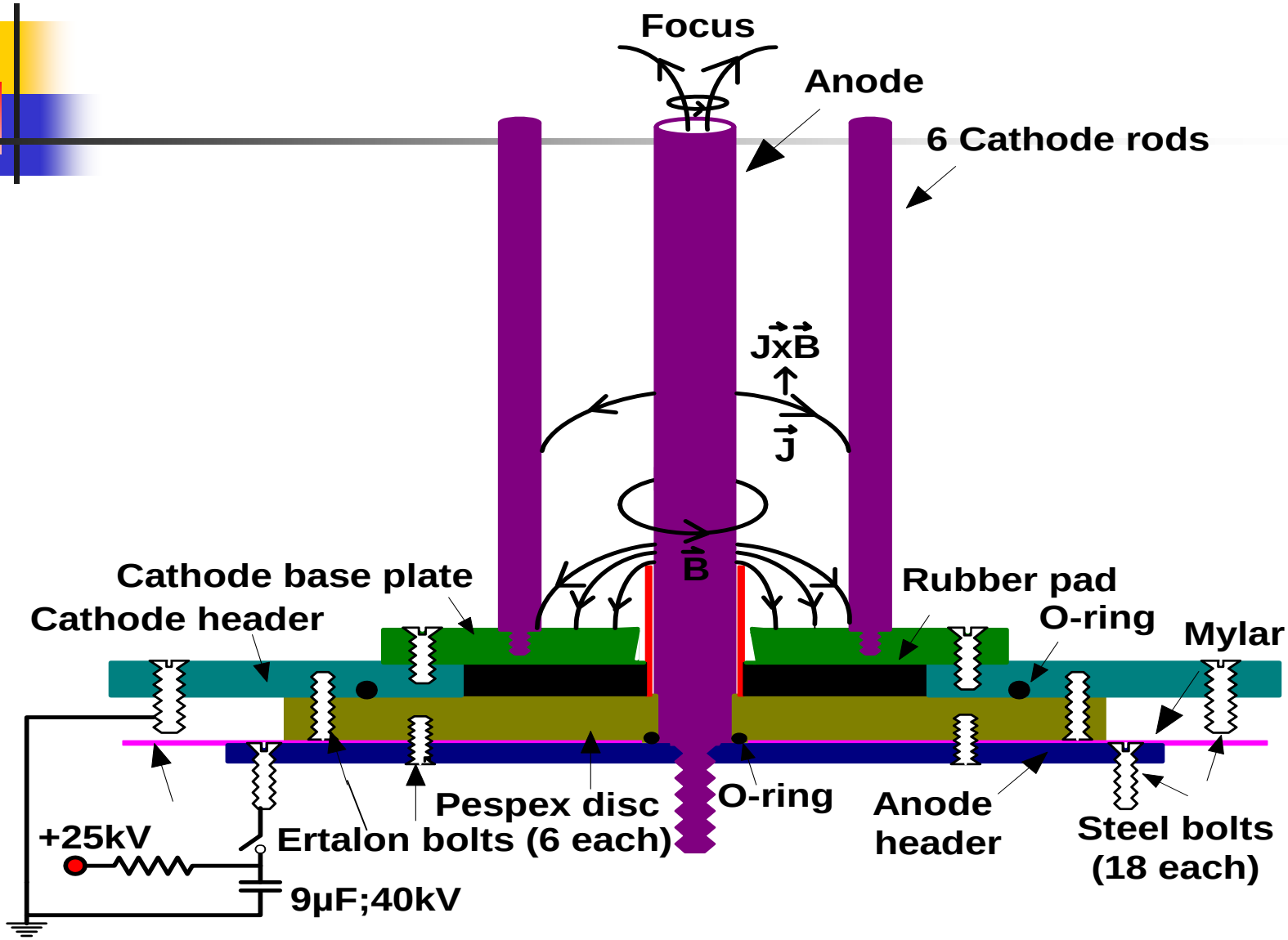
- Carburizing of aluminum however could provide a suitable alternative of nitriding for improved mechanical and tribological properties in a similar way as in the case of iron and steel.
- This inspired to study the ability of plasma focus for carburizing of aluminum.

# Experimental Setup

- The Plasma focus system consists of a coaxial electrode assembly with an anode of aluminium rod having an effective length of 105 mm and a diameter of 18 mm located at the centre surrounded by six equidistant symmetrical copper rods forming cathode with cathode to anode radius ratio of 3.2. The anode is slightly tapered towards the open end to enhance the emission of charge particles.

- A Pyrex glass insulator sleeve of 23 mm breakdown length, with an external diameter of 24.5 mm and wall thickness of 2 mm, is used to separate the anode from the cathode base plate. A field distortion type spark gap is used to transfer energy from the capacitor bank to the electrodes of plasma focus system. The system is operated in a gas flow mode to get rid of the impurities.

# ELECTRODES ASSEMBLY OF DPF



- Good focussing is obtained at 0.7 mbar of methane at 18kV charging voltage.
- These values of charging voltage and optimum filling pressure are maintained throughout the experiment.

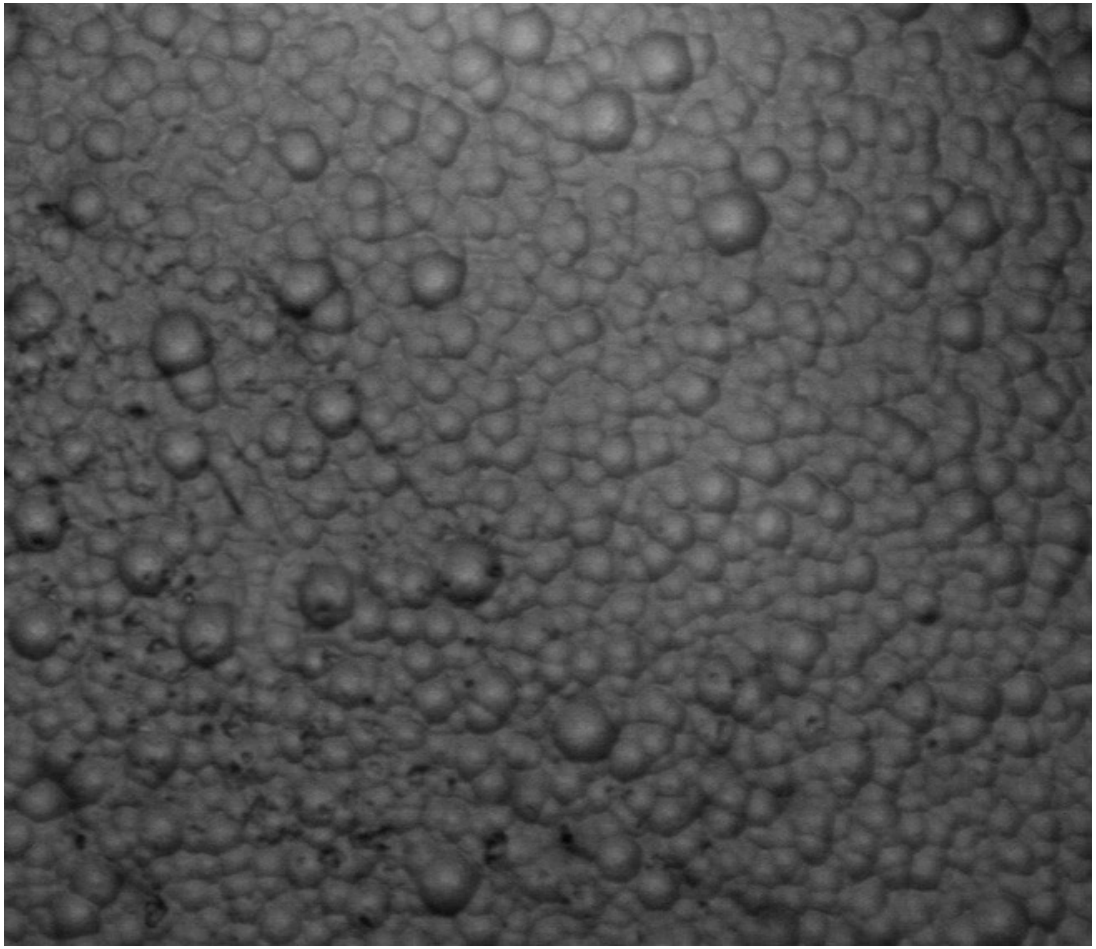
- The aluminium specimens of size 10×10×03 mm are polished mechanically to a mirror finish prior to position at an axial distance of 8 cm from the anode tip.
- An aluminium shutter (covering the specimen) between the ion source and specimen is used to avoid the effects of ion beams emitted during the conditioning shots.

# Diagnostics

- A fast response photoconductive GaAs detector masked with a 50 μm diameter pinhole is employed at a distance 8 cm in front of the anode tip to measure the energy of the ions emitted from the plasma focus.
- The spatial distribution of the ion beam reaching the specimen is obtained by placing a CR-39 track detector at a distance of 8 cm in front of the central anode tip.

- The performance of the plasma focus device i.e. sharp focusing and energetic ion emission is most crucial in these type of experiments. Thus signals from the GaAs detector, voltage probe and Rogowski coil is constantly monitored on Gould 4074A four channel digital storage oscilloscope.

# Results



# Results

- The energy of the ions are found to be in the range of approximately 20-300 keV.
- The tracks shown in the figure are the result of six hours etching of detector in a 6N-NaOH aqueous solution at 70 $^{o}$C. The figure reveals that distribution of ions is mostly uniform. It can be observed that several populations of ions with different energy values are also present. The track density is found to vary from about $7\times10^{4}$ tracks mm$^{-2}$ for 0° direction to $6.2\times10^{4}$ tracks mm$^{-2}$ for $\pm$ 20° direction.

# Raman Spectroscopic results

- Raman spectroscopic measurements of carbon ions implanted samples are performed in a back scattering geometry using an Avantes Raman microprobe equipped with 785 nm line of diode laser.
- The spectra provide information about the Al-C and Al-O bonds.
- The Al-C bond stretching vibrational mode is present at about 760 $cm^{-1}$ and the peaks of Al-O at around 570 and 850 $cm^{-1}$.
- The C-C clustering is monitored by D (around 1330-1350) and G peaks (1100-1700 $cm^{-1}$).

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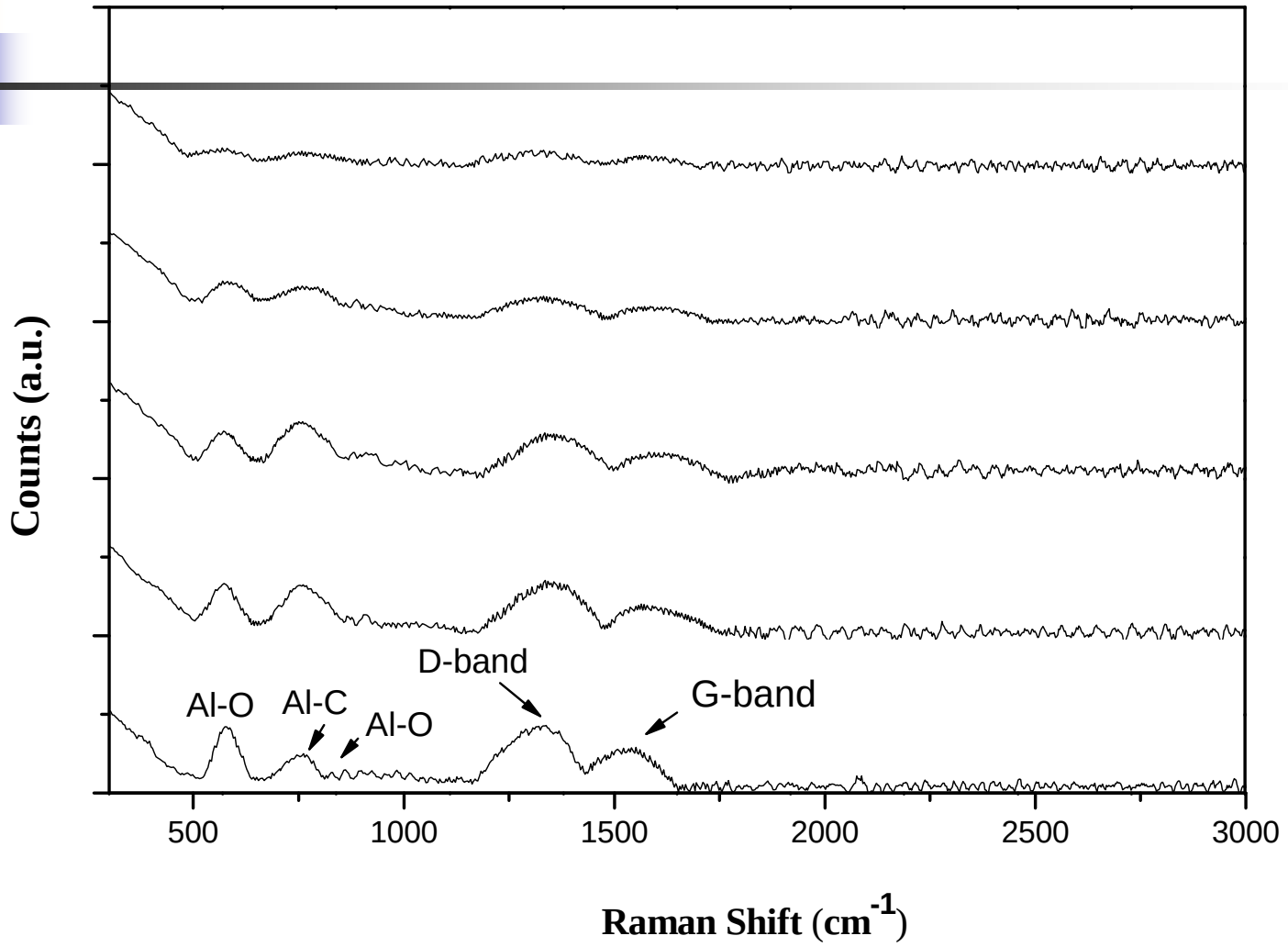
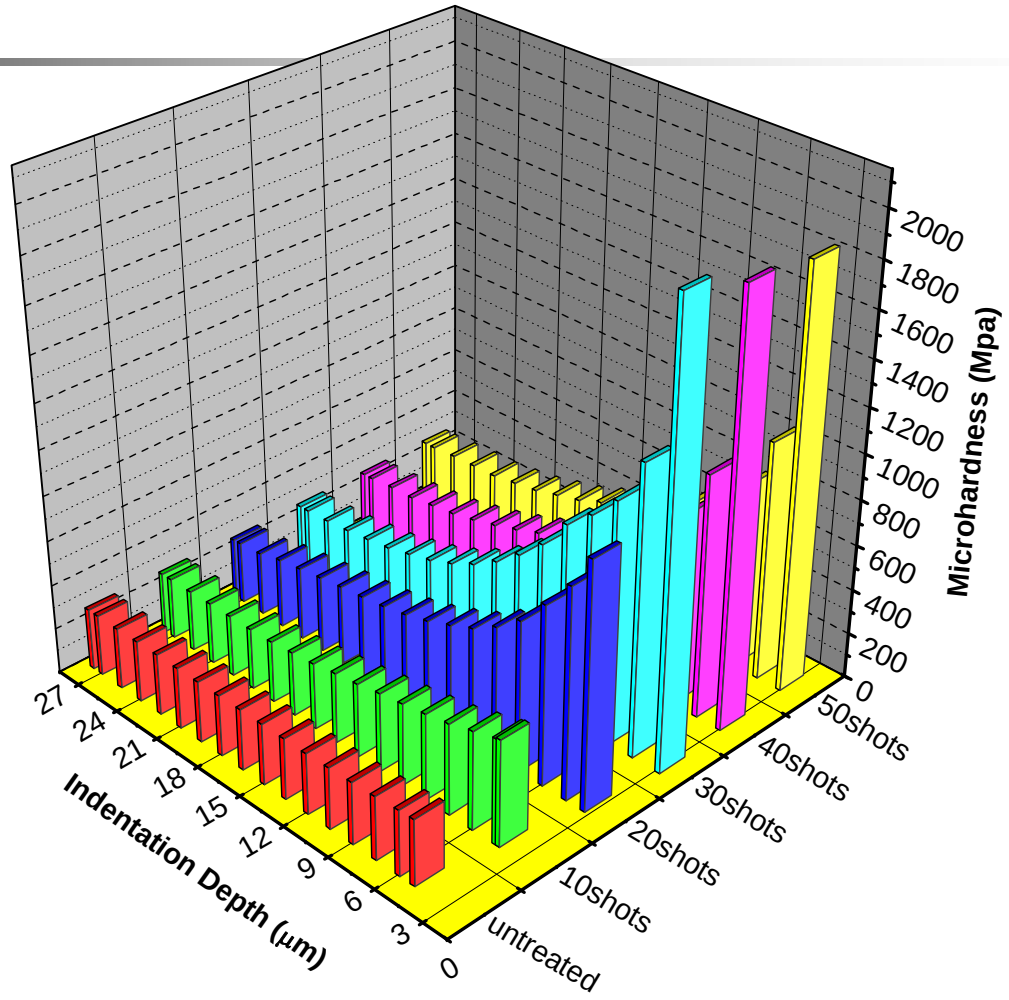

Counts (a.u.)
Al-O
Al-C
Al-O
D-band
G-band
500
1000
1500
2000
2500
3000
Raman Shift (cm-1)

- It is considered that the carbon present in the aluminum matrix as C-C cluster, as result of initial focus shots, may convert to aluminum carbide phase by the preceding ion dose.

# Hardness results

- The surface hardness as a function of the indentation depth is measured with Vickers micro hardness tester. Different loads (10-500 g) are applied to obtain micro hardness.


Microhardness (Mpa)
2000
1800
1600
1400
1200
1000
800
600
400
200
0
Indentation Depth (μm)
27
24
21
18
15
12
9
6
3
0
untreated
10shots
20shots
30shots
40shots
50shots

- The surface hardness increases continuously with increasing ion dose upto 30 focus shots and reaches to a maximum of 1990 MPa, which is nearly a 7 times as compare to the aluminum bulk value.
- The enhanced surface hardness can be attributed to the formation of $Al_4C_3$ phase and amorphous carbon. A slight decrease in surface hardness is observed.
- When the samples are treated with more than 30 focus shots, a decrease in surface hardness is observed. This may be due to poor crystalline phase formation and damage of pre-formed carbide phase by the enhanced ions irradiation.

# SEM results

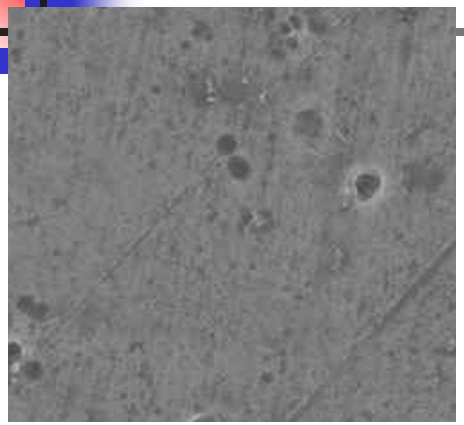
- The surface morphology of pristine and plasma carburized samples is studied by obtaining micrographs using a JEOL JSM-5910 scanning electron microscope (SEM).
- No significant features except the fine scratches induced by polishing are observed for a pristine sample.

- The surface smoothness seems to increase with increasing number of focus shots upto 30.
- It is assumed that upto 30 focus shots, in addition to $Al_4C_3$ phase and C-C clusters formation, the energy density of ions is suitable to remove certain voids, asperities and rough particles by sputtering and etching processes and a smooth carburized surface is achieved.

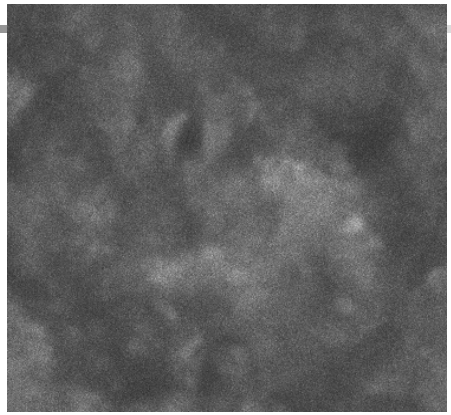
- When the number of focus shots reaches to 40, surface layer becomes slightly rough with wavy wrinkle-like structure, which represents a thin superficially molten layer of the substrate. High doses of carbon and unbound carbon result in surface swelling of substrate increasing its roughness as in the case of nitriding.

- Surface treated with 50 focus shots shows rough surface and some micro-cracks with low density small pores. It is assumed that high fluences of ion, generated by stable and strong focusing action, deliver excessive energy to substrate, causing sputtering and etching and hence resulted in surface damage and roughness along with a very few small pores.

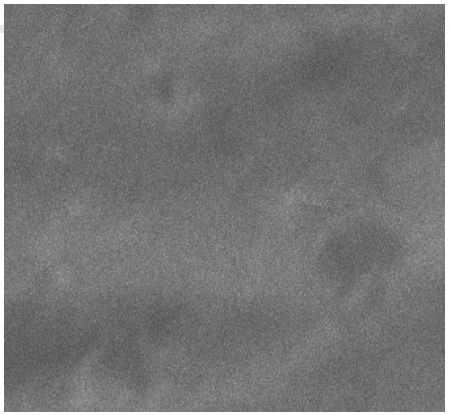
- The quenching after the transient temperature rise with short pulse ion beam resulting in the brittleness of the sample may be the reason behind the development of micro cracks.



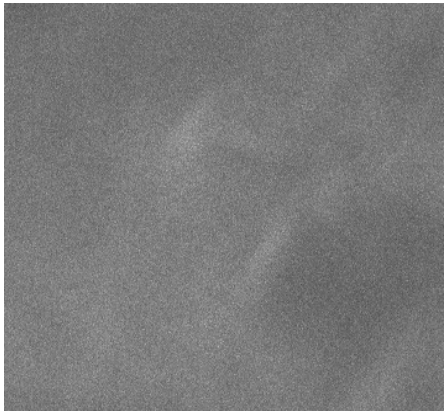
Untreated



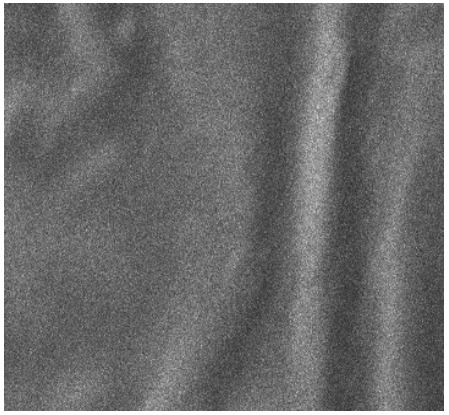
10 Shots



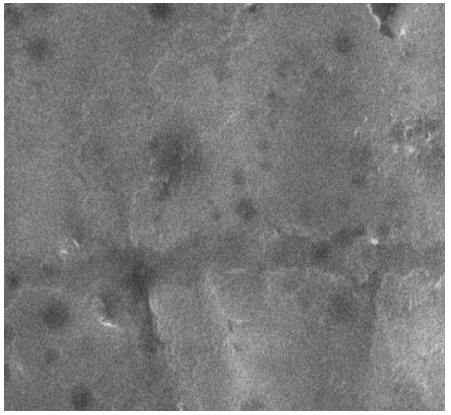
20 shots



30 shots



40 shots



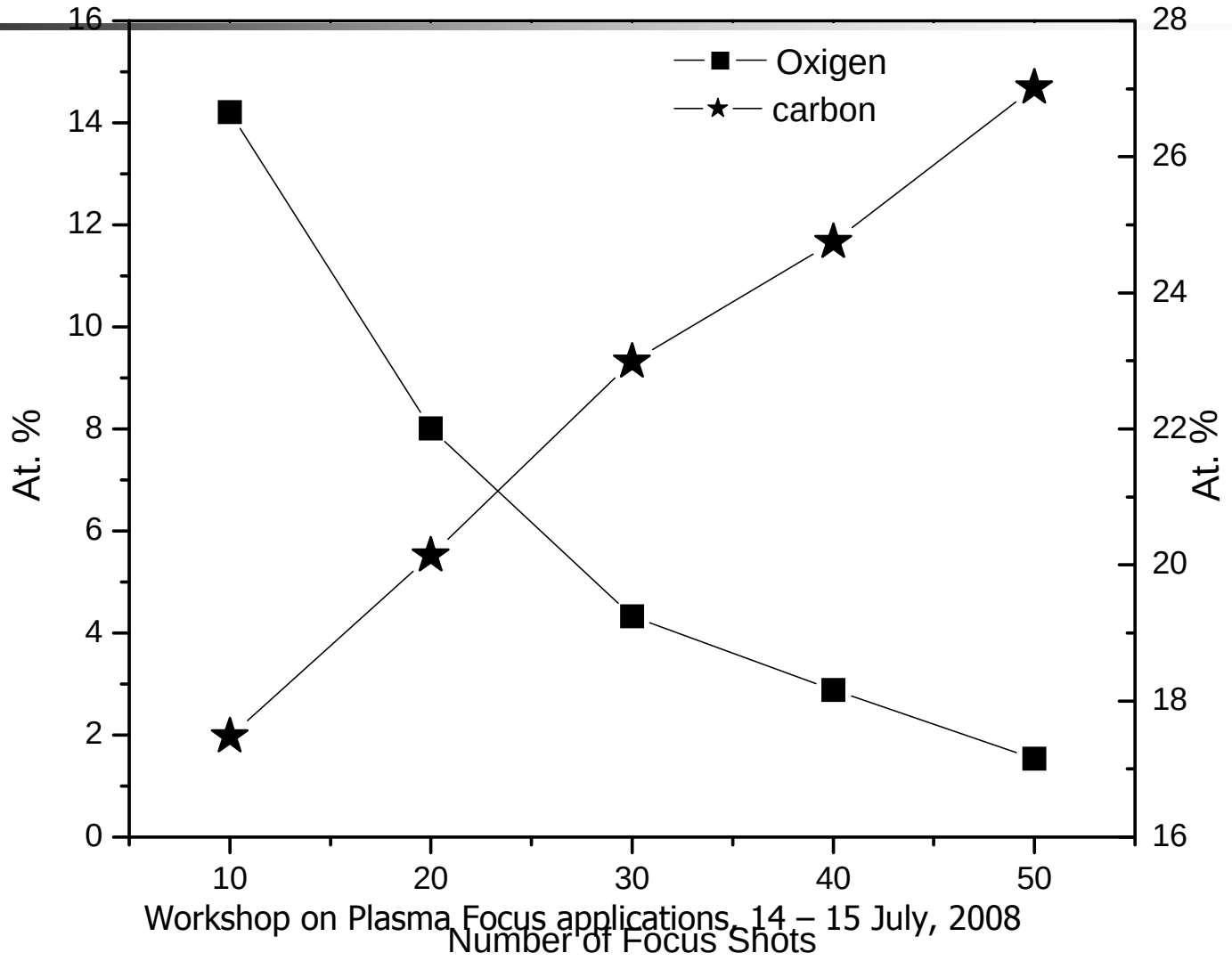
50 shots

# EDX Results

- The variation of carbon and oxygen concentration with increasing number of focus shots is obtained by EDX attached with the SEM .
- The oxygen peak in the untreated sample is present because of native oxide layer.

- The concentration of oxygen is reduced by ion sputtering process with increasing number of focus shots but a negligible amount remains present. This may be due to re-oxidation of near surface region by residual gas and by the process of ion beam mixing. The concentration of carbon increases with increasing number of focus shots as expected.

# EDX


Oxigen
carbon
At. %
At. %
Number of Focus Shots
0
2
4
6
8
10
12
14
16
16
18
20
22
24
26
28
10
20
30
40
50

# CONCLUSIONS

- Carburizing of aluminum surface is achieved with methane plasma ion implantation by using a Mather type plasma focus device over a multiple focus shot.
- These results suggest the parameters for plasma focus device to get a quality carburized aluminum surface.

- Raman spectroscopic results indicate that ion fluences upto 30 focus shots are suitable to develop $Al_4C_3$.
- Raman spectra also reveal the presence of diamond type tetragonal $sp^3$-bounded carbon (D-band) and graphite type trigonal $sp^2$-bounded carbon (G-band) in the implanted surface
- Approximately seven times improved surface hardness is attributed to the hexagonal $Al_4C_3$ phase, $sp^2$ and $sp^3$-bounded carbon content.

- The SEM results reveal the surface smoothness dependence on the energy density of ions.
- The energy of ion flux delivered by 30 focus shots is found optimum to get smooth and mechanically applicable carburized aluminium surface.
- The reduction in oxygen concentration upto a certain value with increasing number of focus shots dictate the ability of plasma focus to remove native oxide layer.

- Based on these investigations, the plasma focus device is found an efficient device to get a carburized aluminum surface at room temperature environment in lesser time.