Plasma Cleaner

Last Updated: 4/20/2020

You must be a “Qualified Self-User” to operate this instrument independently.
You must be on the labs “Instrument Reservation Schedule” before touching the instrument for any reason.
Any problems, STOP, Post a note on the instrument and send an email to mtim@mit.edu immediately.
Do not perform any maintenance.
Do not install any software
Do not adjust any optics.

Instrument Hazards:
Flammable Gas (Oxygen), Gas Cylinder & Electricity.

Important:
To prevent oil from back streaming from the mechanical pump into the process chamber,
Do not let the vacuum in the chamber go below 100um!

Required Apparel:
Safety Glasses
Clean Gloves
**Find System:**

- Plasma Cleaner
  - Power On
  - Vacuum Off
  - RF Off
- GasFlo Unit
  - Power On
  - O2 & N2 Flow valves closed

**Utilities**

- Nitrogen (Supplied from two Liquid Nitrogen Tanks in hallway)
- Oxygen Gas Cylinder
- Electricity (110VAC always on)
- Exhaust (Mechanical pump vents to the exhaust hood).

**Operation:**

**Prepare the chamber**

- Hold the lid onto the vacuum chamber.
- Turn on the mechanical pump.
- Wait until the pressure drops below 500mtorr.
- Open the Oxygen flowmeter valve and stabilize the pressure at ~800-1000mtorr.
- Turn on the RF (High).
- Verify you see a Plasma glowing in the chamber by looking through the vents on the side.
- Keep the rf on for ~five minutes.
- Do not keep the rf on if there is no plasma (proper pressure must be maintained).
- Do not keep the plasma on for more than 10 minutes at a time (heats up).
- Turn off the RF power.
- Close the Oxygen flow meter valve and wait for the pressure to get to ~200mtorr.
- Turn off the mechanical pump.
- Hold onto the chamber lid to prevent it from falling when the chamber is vented.
- Open the Nitrogen flowmeter valve (~1/2 turn).
- Wait until the chamber lid is loose.

**Clean your sample**

- Place your sample in the chamber.
- Hold the lid onto the vacuum chamber.
- Turn on the mechanical pump.
- Close the Nitrogen Flowmeter Valve.
- Wait until the pressure drops below 500mtorr.
- Open the Oxygen or Nitrogen flowmeter valve and stabilize the pressure at ~800-1000mtorr.
- Turn on the RF (High).
- Verify you see a Plasma glowing in the chamber by looking through the vents on the side.
Keep the rf on for ~five minutes.
Do not keep the rf on if there is no plasma (proper pressure must be maintained).
Do not keep the plasma on for more than 10 minutes at a time (heats up).
Turn off the RF power.
Close the Oxygen flow meter valve and wait for the pressure to get to ~200mtorr.

Pump & Purge so Oxygen is not left standing in the mechanical pump
Turn on the Nitrogen gasflow valve ½ turn for ~5 seconds.
Close the Nitrogen gasflow valve.
Wait for the pressure to go below ~300mtorr.
Turn on the Nitrogen gasflow valve ½ turn for ~5 seconds.
Close the Nitrogen gasflow valve.
Wait for the pressure to go to ~300mtorr.

Turn off the mechanical pump.
Hold onto the chamber lid to prevent it from falling when the chamber is vented.
Open the Nitrogen flowmeter valve (~1/2 turn).
Wait until the chamber lid is loose.

**Shutdown after use:**

  Plasma Cleaner
    Power On
    Vacuum Off
    RF Off
  GasFlo Unit
    Power On
    O2 & N2 Flow valves closed

Don’t forget to disengage CORAL!
Utilities:
Electricity (wall).
Nitrogen from bulk liquid nitrogen dewars in hallway.
Oxygen gas cylinder.
Exhaust Hood for mechanical pump exhaust.

Emergency Shutdown:
Turn the outlet strip power off.
Close the oxygen gas cylinder valve.
Close the liquid nitrogen dewar valves (Hallway).

Restart after an emergency:
Turn the outlet strip power on.
Open the oxygen gas cylinder valve.
Open the liquid nitrogen dewar valves (Hallway).
Specifics:
Harrick Plasma
Model PDC-32G
Input Power 100W
Applied to the RF Coil
Low Setting 680V DC, 10 mA DC, 6.8W
Medium Setting 700V DC, 15 mA DC, 10.5W
High Setting 720V DC, 25 mA DC, 18W

Features
Compact, tabletop unit
Adjustable RF power
Low, Medium, and High power settings
PDC-32G (115V)
Basic model
Includes a 3" diameter x 6.5" length Pyrex chamber and a removable front cover assembly
Applies a maximum of 18W to the RF coil
1/8" NPT needle valve to qualitatively control gas flow and chamber pressure
1/8" NPT 3-way valve to quickly switch from bleeding in gas, isolating the chamber, and venting
Weight: 13 lbs
Size: 8.5" H x 10" W x 8" D
Optional PlasmaFlo gas mixer allows quantitative control of up to two (2) process gases and monitoring of chamber pressure
Optional quartz chamber and sample tray

Info from Manufacturers website:
http://www.harrickplasma.com/products_cleaners.php

Contact:
info@harrickplasma.com • 800.640.6380 (US&CN) • 607.272.5070 (Intl)
**Principle of Operation**

When a gas under sufficiently low pressure is subjected to a high frequency oscillating electromagnetic fields, the accelerated ions in the gas collide with the gas molecules ionizing them and forming a plasma.

The ionized gas particles in the plasma interact with solid surfaces placed in the same environment by:

- Removing organic contamination from the surfaces.
- The high energy plasma particles combine with the contaminant to form carbon dioxide (CO2) or methane (CH4).

Modifying or enhancing the physical and chemical characteristics of surfaces.
A chemical reaction occurs between the plasma gas molecules and the surface undergoing treatment.

The sample is placed in the reaction chamber. Low flow rates (5-10SCFH) of a process gas at low pressure (200-600mtorr) are subjected to Radio Frequency (RF) electromagnetic radiation at 8-12 MHz creating plasma within the chamber.

The type of interaction between the plasma and the surface depends on parameters such as the intensity and frequency of the RF power used to excite the plasma, the type of gases that are ionized, the pressure and flow rate of the gases, the type of sample and the amount of time the surface is exposed to the plasma.
Applications
http://www.harrickplasma.com/applications.php

Surface Cleaning and Surface Treatment
Harrick Plasma Cleaner models may be employed in a variety of application fields, including materials science, polymer science, biomedical materials, microfluidics, optics, microscopy and dental & medical research.

Links to further information on plasma surface cleaning and surface treatment with Harrick Plasma Cleaner models are given below:

**Plasma Cleaning**
Benefits of plasma cleaning for surface preparation prior to bonding and other applications; plasma processing guidelines and suggestions; contact angle measurements on plasma-treated glass showing enhanced surface wettability.

**Microfluidic Devices**
Benefits of plasma treatment for microfluidic device fabrication; plasma processing guidelines and suggestions for plasma treatment of PDMS prior to bonding; contact angle measurements on plasma-treated PDMS.

**Surface Activation and Modification**
Benefits of plasma treatment for modifying surface chemistry through restructuring, grafting, or deposition; plasma processing guidelines and suggestions; examples of surface chemistry and contact angle measurements on plasma-treated polymers.

**Surface Adhesion and Wettability**
Benefits of plasma treatment in altering surface wettability characteristics for adhesion and other applications; plasma processing guidelines and suggestions; examples of contact angle measurements on plasma-treated materials.

**Biomaterials**
Benefits of plasma treatment for biomaterials through modification of surface wetting properties and surface sterilization; plasma processing guidelines and suggestions.

**PLASMA APPLICATIONS:**

**Plasma Cleaning**
For references citing the use of our plasma cleaners, categorized by research application, see the References: Technical Articles page.

**Benefits of Plasma Cleaning**
Remove organic contaminants by chemical reaction (O\textsubscript{2} or air plasma) or physical ablation (argon plasma)
Eliminate the use of chemical solvents as well as storage and disposal of solvent waste
Clean surfaces with microscale porosity or microchannels not suitable for solvent cleaning due to surface tension limitations
Render most surfaces hydrophilic; decrease water droplet contact angle and increase surface wettability [Figure 1] (see also Surface Adhesion and Wettability)
Promote adhesion and enhance bonding to other surfaces
Prepare surface for subsequent processing (e.g. film deposition or adsorption of molecules)
Sterilize and remove microbial contaminants on the surface; beneficial for biomedical applications and biomaterials (see also Biomaterials)
Clean surface without affecting the bulk properties of the material
Can treat a wide variety of materials as well as complex surface geometries; examples include:
- Semiconductor wafers and substrates (Si, Ge)
- Glass slides and substrates
- Optics and optical fibers
- Oxides (quartz, indium tin oxide (ITO), TiO\textsubscript{2}, Al\textsubscript{2}O\textsubscript{3}); mica
- Gold and metal surfaces
- Electron microscopy (EM) grids
- Atomic force microscopy (AFM) cantilever tips

**Applications**
Clean substrates to reduce background autofluorescence originating from organic contaminants for fluorescence microscopy
Clean optics, crystals (quartz, Ge, ZnSe), cuvettes, and substrates for spectroscopic measurements (ATR-FTIR, UV-Vis, SERS)
Clean quartz crystals for quartz crystal microbalance (QCM) measurements
Clean AFM cantilever tips for surface morphology and frictional force measurements
Clean electron microscopy (EM) grids, specimen holders, and substrates
Clean printed circuit (PC) board and die surface prior to bonding
Clean gold surfaces for self-assembly experiments

**Processing Methods**
Oxygen or air plasma
Removes organic contaminants by chemical reaction with highly reactive oxygen radicals and ablation by energetic oxygen ions
Promotes hydroxylation (OH groups) on the surface
May oxidize the surface; oxidation may be undesirable for some materials (e.g. gold) and may affect surface properties
Argon plasma
Cleans by ion bombardment and physical ablation of contaminants off the surface
Does not react with the surface or alter surface chemistry
For applications that are sensitive to potential contamination from trace impurities (e.g. Ca, K, Na) in borosilicate glass, a quartz chamber is recommended over the standard Pyrex chamber
Suggested process parameters values for plasma cleaning using a Harrick Plasma cleaner (some experimentation may be required to determine optimal process conditions)
Pressure: 100 mTorr to 1 Torr
RF power: Medium or High
Process time: 1-3 minutes
Low RF power may be used to minimize surface roughening; the process time may require adjustment to compensate for the lower power.


PLASMA APPLICATIONS:
Microfluidic Devices
For references citing the use of our plasma cleaners in microfluidic applications, see the Microfluidic Devices link to category of the References: Technical Articles page.

Benefits of Plasma Treatment for Microfluidic Device Fabrication
Quickly render poly(dimethylsiloxane) (PDMS), glass, and other polymer surfaces hydrophilic through plasma oxidation [Figure 1]
Bond oxidized PDMS surfaces and seal irreversibly to create leak-tight channels in microfluidic devices
Hydrophilic surfaces enhance fluid flow and wetting of channels in microfluidic devices
Pattern surfaces with alternating hydrophilic-hydrophobic regions

Applications
Study of chemical reactions and fluid flow on micron scale
Detection of biological organisms or chemical species
Clinical diagnostics and drug screening for medical research
Manipulation of fluid on cellular length scale (microns) for biological research
Growth of cell and tissue cultures
**Processing Methods**

General processing sequence

Pattern PDMS by replica molding from a master mold

Oxidize PDMS in O$_2$ or air plasma, etching hydrocarbons and leaving silanol (SiOH) groups on the surface, rendering the surface hydrophilic

Place in contact with another oxidized PDMS or glass surface to form bridging Si-O-Si bond at the interface, creating an irreversible seal

Suggested process protocol guidelines and process parameter values for plasma treatment of PDMS using a Harrick Plasma Cleaner (some experimentation may be required to optimize process)

Use oxygen (O$_2$) or room air as the process gas

Pressure: 100 mTorr to 1 Torr

RF power: Medium or High

Process time: 20-60 seconds

An example protocol for PDMS plasma treatment using a Harrick Plasma cleaner as demonstrated in a video published by the Jeon Research Group


Avoid extended plasma treatment times; prolonged plasma exposure causes cracking in PDMS and migration of low molecular mass molecules from bulk to surface, decreasing the number of hydrophilic SiOH groups and resulting in weak or incomplete bonding


Oxidized surfaces should be brought into contact immediately after plasma treatment to achieve strongest bond possible

PDMS surface recovers hydrophobic properties (ages) with time after plasma treatment (~1 hour)


PLASMA APPLICATIONS:
Surface Activation and Modification
For references citing the use of our plasma cleaners for surface activation and modification, see the Surface Modification and Surface Wettability categories in the References: Technical Articles page.

Benefits of Plasma Treatment
Modify surfaces by attachment or adsorption of functional groups to tailor surface properties for specific applications
Restructure polymer surfaces through crosslinking
Deposit polymer layers by plasma polymerization
Graft functional polymers or end groups onto plasma-activated surfaces [Figure 1]
Prepare surfaces for subsequent processing, e.g. film deposition or adsorption of molecules
Improve surface coverage and spreading of coatings and enhance adhesion between two surfaces (See Surface Adhesion and Wettability)
Modify wettability to render a surface hydrophilic [Figure 2] or hydrophobic [Figure 3] with the appropriate process gas(es)
Change surface properties without affecting the bulk material

**Applications**

- Self assembly studies with patterned hydrophilic and hydrophobic surfaces
- Dental research of periodontal cell adhesion
- Promote adhesion of microorganisms on plasma-modified surfaces
- Promote adhesion of cells and cell proliferation on plasma-modified biomaterials or tissue scaffolds
- Modify surface to act as protective or barrier layer to the bulk material
- Crosslink polymer surfaces to reduce permeability of specific molecules
- Render surfaces hydrophilic by oxidation and formation of hydroxyl (OH) groups
- Render surfaces hydrophobic with deposition of fluorine-containing groups (CF, CF₂, CF₃)

**Processing Methods**

- Oxygen or air plasma
  - Removes organic contaminants by chemical reaction with highly reactive oxygen radicals and through ablation by energetic oxygen ions
  - Promotes surface oxidation and hydroxylation (OH groups); increase surface wettability
  - Oxidation may be undesirable for some materials (e.g. gold) and can affect surface properties
- Argon plasma
  - Cleans by ion bombardment and physical ablation of contaminants off the surface
  - Does not react with the surface or alter surface chemistry
- Carbon tetrafluoride (CF₄) plasma
  - Forms hydrophobic coating of fluorine-containing groups (CF, CF₂, CF₃)
  - Decreases number of hydrophilic polar end groups on surface; decreases surface wettability
  - Surfaces should be used immediately after plasma treatment; plasma-treated surfaces may recover their untreated surface characteristics with prolonged exposure to air
- Suggested process parameters values for plasma treatment using a Harrick Plasma cleaner (some experimentation may be required to determine optimal process conditions)
  - Pressure: 100 mTorr to 1 Torr
  - RF power: Medium or High
  - Process time: 1-3 minutes
Figure 1. Surface density of carboxyl (COOH) groups as a function of air plasma treatment time, using a Harrick Plasma cleaner, on 100 μm thick poly(caprolactone) (PCL) nanofiber mats. The COOH layer facilitates subsequent grafting of gelatin molecules onto the PCL nanofiber mats for potential use as tissue-engineering scaffolds. Data from Ma, Z., W. He, T. Yong, S. Ramakrishna. "Grafting of Gelatin on Electrospun Poly(caprolactone) Nanofibers to Improve Endothelial Cell Spreading and Proliferation and to Control Cell Orientation." Tissue Eng. (2005) 11: 1149-1158.

PLASMA APPLICATIONS:
Surface Adhesion and Wettability

For references citing the use of our plasma cleaners in adhesion and wettability applications, see the Surface Adhesion and Surface Wettability categories in the References: Technical Articles page.

Benefits of Plasma Treatment

Remove residual organic impurities and weakly bound organic contamination
Prepare surfaces for subsequent processing (e.g. film deposition or adsorption of molecules)
Improve surface coverage and spreading of coatings and enhance adhesion between two surfaces
Modify wettability to render a surface hydrophilic [Figure 1 and Figure 2] or hydrophobic [Figure 3] with the appropriate process gas(es)
Affect only a few monolayers of the surface; does not change bulk properties of the material
Can treat a wide variety of materials as well as complex surface geometries; examples include:
Semiconductor wafers and substrates (Si, Ge)
Glass slides and substrates
Oxides (quartz, indium tin oxide (ITO), TiO$_2$, Al$_2$O$_3$); mica
Polymers (PE, PDMS, PEEK, PTFE, PLA)
Metal surfaces (gold, stainless steel)
Electron microscopy (EM) grids

Applications
Surface preparation of substrates prior to self-assembly experiments
Surface preparation of electron microscopy (EM) sample grids
Plasma cleaning of printed circuit (PC) boards and die surfaces prior to bonding
Plasma treatment of dental implant and impression mold materials
Plasma treatment of biomaterials and biomedical devices prior to functionalizing surface
Plasma treatment of fibers to improve adhesion to matrix in fiber-reinforced composite materials
Study of adhesion characteristics of dissimilar materials by mechanical testing or atomic force microscopy (AFM) force measurements

**Processing Methods**

Oxygen or air plasma
Removes organic contaminants by chemical reaction with highly reactive oxygen radicals and ablation by energetic oxygen ions
Promotes surface oxidation and hydroxylation (OH groups); increase surface wettability
Oxidation may be undesirable for some materials (e.g. gold) and can affect surface properties

Argon plasma
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Carbon tetrafluoride (CF₄) plasma
Forms hydrophobic coating of fluorine-containing groups (CF, CF₂, CF₃)
Decreases number of hydrophilic polar end groups on surface; decreases surface wettability
Surfaces should be used immediately after plasma treatment; plasma-treated surfaces may recover their untreated surface characteristics with prolonged exposure to air

Suggested process parameters values for plasma treatment using a Harrick Plasma cleaner (some experimentation may be required to determine optimal process conditions)
Pressure: 100 mTorr to 1 Torr
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![Surface Modification of 316L Stainless Steel](image)

*Figure 1. Water drop contact angle measurement on 316L stainless steel (a) as received, (b) after chemical clean (ultrasonication in 70% ethanol, acetone, and 40% nitric acid), and (c) after chemical clean and O₂ plasma treatment using a Harrick Plasma cleaner. Data from Mahapatro, A., D. M. Johnson, D. N. Patel, M. D. Feldman, A. A. Ayon, C.*


PLASMA APPLICATIONS:

Biomaterials

For references citing the use of our plasma cleaners in biomaterials applications, see the Biology, Biomedical Applications, and Cleaning and Sterilization categories in the References: Technical Articles page.

Benefits of Plasma Treatment

Enhance adhesion and modify surface wetting properties of biomaterials

Biomaterials are typically chemically inert and have low surface energies to minimize fouling and undesired interaction with other surfaces; these properties also make it difficult to effectively apply functional coatings or active molecular groups on the surface

Plasma treatment can render surfaces hydrophilic to improve adhesion for subsequent coating or adsorption of functional groups, or render surfaces hydrophobic through deposition of fluorinated end groups

Plasma treatment can enhance functionality and biocompatibility of biomaterial surfaces

Sterilization

Oxygen plasma treatment can simultaneously clean and surface sterilize medical devices and biomaterials
Plasma sterilization is appropriate for medical or dental implants and devices that are sensitive to the high temperature, chemical or irradiative environments associated with autoclaving, ethylene oxide (EtO) or gamma sterilization, respectively.

**Applications**

Plasma activation of substrate surfaces to render surface hydrophilic; promote attachment and adhesion of functional biological species or coatings

Enhance cell adhesion, coverage, and proliferation on tissue scaffolds

Promote adsorption of selective functional biological species while resisting adhesion of bacteria and fouling microorganisms

Apply coatings on plasma-treated biomaterial surfaces to act as a protective barrier layer or lubricant in implanted medical devices

Plasma cleaning and activation of microelectrode arrays for biosensors

Sterilization of medical devices and biomaterials (e.g. dental implants, dental impression mold materials, tissue scaffolds)

**Processing Methods**

Oxygen or air plasma

Removes organic contaminants by chemical reaction with highly reactive oxygen radicals and ablation by energetic oxygen ions

Promotes surface oxidation and hydroxylation (OH groups); increases surface wettability

Oxidation may be undesirable for some materials (e.g. gold) and can affect surface properties

Argon plasma

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Decreases number of polar end groups on surface; decreases surface wettability

For applications that are sensitive to potential contamination from trace impurities (e.g. Ca, K, Na) in borosilicate glass, a quartz chamber is recommended over the standard Pyrex chamber

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