



Plasma generation by household microwave oven for surface modification and other emerging applications

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A simple and inexpensive method to generate plasma using a kitchen microwave oven is described in this paper. The microwave-generated plasma is characterized by spectroscopic analysis and compared with the absorption spectra of a gas discharge tube. A Paschen-like curve is observed as the microwave plasma initiation time is plotted as a function of the pressure of the plasma chamber. We have also demonstrated that this microwave-generated air plasma can be used in a multitude of applications such as: (a) surface modification of a substrate to change its wettability; (b) surface modification to change electrical/optical properties of a substrate; and (c) enhancement of adhesive forces for improved bonding of polymeric microfluidic molds, such as bonding polydimethylsiloxane (PDMS) chips to glass covers. These simple techniques of plasma generation and subsequent surface treatment and modification applications may bring new opportunities leading to new innovations not only in advanced labs, but also in undergraduate and even high school research labs. © 2021 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>).

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I. INTRODUCTION

Plasma is ubiquitous in nature and readily observed as lightning on a stormy night, or as a static electric spark the moment before touching a door knob on a dry winter day.¹ It is also found naturally occurring in stars,² solar winds,³ upper atmospheric lighting,⁴ and the awe-generating lightning glow known as St. Elmo's fire⁵ that sailors wondered about for centuries. Practical applications of plasma span a vast domain including, but not limited to, fusion energy generation,^{6–9} plasma TV displays,¹⁰ plasma enhanced chemical vapor deposition (PECVD),¹¹ sputtering,¹² semiconductor device fabrication,^{13,14} substrate cleaning,¹⁵ and

sterilization.¹⁶ Plasma is also referred to as the fourth state of matter.¹⁷ As the addition of energy to material in a solid or first state initiates a phase change to a liquid second state and then to a gaseous third state, adding a sufficient amount of energy to a gas may ionize the gas by ejecting electrons from the outer shells and/or through collisions of individual ions. Plasma, the fourth state,¹⁷ is thus composed of a conducting soup of energized charged particles, electrons, and ionized atoms/molecules that are normally generated by the application of strong electromagnetic or heat energy. Once this highly charged gas is brought in contact with a surface, some energy is transferred from the plasma to the substrate surface, in turn changing the surface property of the substrate.



Unfortunately, plasma instrumentation remains somewhat sophisticated and expensive, preventing its widespread use in teaching and research environments for institutions with small budgets. In the past, some attempts were made to bring plasma research and analysis to high school and undergraduate teaching and research labs;^{18–20} however, they involve either high temperature flames²¹ or gas discharge tubes that require high voltage and vacuum levels for the proposed experiments, raising potential safety concerns and related issues in elementary labs. Beck *et al.*²² also computed trajectories of electrons in a weakly coupled plasma obtained from particle simulations. However, the goal of nearly all of these earlier theoretical or experimental papers for undergraduate students was to characterize plasma and experimentally/numerically verify the theory of plasmas. In a recent paper,²³ the popular science activity of forming plasma in a microwave with grapes showed that grapes act as spheres of water, which, due to their large index of refraction and small absorptivity, form leaky resonators at 2.4 GHz. When brought together, Mie resonances in isolated spheres coherently add up so that the aqueous dimer displays an intense hotspot at the point of contact. When this hotspot is sufficient to field-ionize available sodium and potassium ions, it ignites a plasma.

In contrast to the previous works, here we show that a simple plasma treatment apparatus can be constructed from an inexpensive household microwave oven and a modified vacuum flask and show many modern applications of this microwave-generated plasma. Although some previous works have shown that plasma can be sparked when a gas held at low pressure is subjected to microwave radiation,^{24–35} this technique has never received the attention it deserves when it comes to a low-cost laboratory technique for a wide range of surface treatments. We demonstrate a number of cutting-edge applications of the plasma device that we have constructed which includes the modification of opto-electrical properties of semiconductors, changing wettability of substrates, enhancement of bonding between certain polymers such as PDMS and glass, and reduction of graphene oxide to graphene for electronics purposes, etc.

Before going into the details of the microwave-generated plasma generation process, we believe a comprehensive discussion about past works on microwave breakdown of air, including similarities and differences between previously observed breakdown phenomena and our plasma generation observation, is necessary to put our work in proper perspective. Breakdown of plasma in air has been studied by many authors. In general, the plasma generation process under DC conditions is relatively well understood, where avalanche multiplication of electrons in a neutral gas is produced through electron impact ionization (or photoionization) when electrons or photons with sufficient energy collide with neutral atoms and molecules present in the medium.³⁶ However, under the influence of an oscillating electric field instead of a DC field, the ions and electrons oscillate back and forth in between the electrodes, and for a very high frequency only a very small number of charged particles are able to reach the boundary electrodes, reducing the diffusive loss of electron energy.

The first serious investigation of microwave breakdown in air was conducted by Herlin and Brown.³² The theoretical basis of their analysis consists of solving the continuity equation for the electron number density in their custom-made cavity geometry, accounting for the source of electrons as impact ionization and loss terms by diffusion and attachment to the gas in use. Breakdown in air in a wide range of conditions

occurs when the rate at which electrons are produced by ionization exceeds the rate at which they are lost by diffusion from the plasma region or by attachment to neutral molecules and atoms. Their custom-made microwave source was a continuous-wave tunable magnetron that operated at about 3 GHz. In order to isolate the microscopic phenomena leading to breakdown they used specifically designed cavities (i.e., cavities that resonated at the TM_{010} mode) so that gas pressure and electric field strength were at all times well-known. A radioactive source was placed near the discharge that provided a small amount of ionization in the cavity to start with, and the microwave field in the cavity was increased until the cavity suddenly began to glow (at which time the microwave field abruptly decreased). The radioactive source in their case was the supplier of the triggering electrons. They found that the breakdown field was smaller at about 3 Torr than it was at higher or lower pressures under these conditions. This minimum also appeared to be a weak function of cavity size. They also found that the dominant mechanism for breakdown of a low pressure gas at microwave frequencies is ionization by collision of electrons with neutral gas molecules rather than loss by diffusion to the walls of the discharge tube.

Afterwards considerable research was done using fundamentally the same apparatus. Gould and Roberts³³ also used a cavity resonated at the TM_{010} mode and a 85 millicurie, cobalt 60 radioactive source was placed on the cavity in order to ensure the presence of sufficient number of electrons in the discharge region even before the microwave source was turned on. They extended the theory to include an additional loss mechanism for electrons: the attachment of electrons to neutral molecules, creating negatively charged molecules. (Of note also is that recombination, the coalescence of an electron with an ion to form an atom, is negligible.)

MacDonald, Gaskell, and Gitterman³⁴ extended the range of frequencies studied and investigated air, nitrogen, and oxygen independently. With the exception of some X-band cavities, all of their cavities were also cylindrical and resonant in the TM_{010} mode. They also reported the necessity of having a 5 mCi cobalt 60 gamma-ray source next to the cavities to produce sufficient ionization to get repeatable results for pulsed waves and a 5 μ Ci source for continuous wave process. Their measurements agreed with previous measurements. They critiqued the previous theory by noting it assumed an electron-molecule collision frequency that was independent of electron energy, when it had long been known that the electron-nitrogen collision frequency was in fact strongly dependent on electron energy. While unable to present an improved analytic theory, they were able to present an alternative representation that allowed an empirical method of calculating breakdown fields in a very large variety of conditions.

MacDonald, Gaskell, and Gitterman's work was extended to form the final chapter (and the [Appendix](#)) of MacDonald's book.³⁰ As had been the case in Herlin and Brown's work, in order to achieve reproducible results a radioactive source (5 mCi cobalt 60) was placed again near the cavity in this work. Bandel and MacDonald³⁵ further measured breakdown electric fields in air, H_2O , and air plus H_2O at 3.06 GHz. In their case, triggering electrons for the discharge were supplied not by a radioactive source, but by photo-electric emission by ultraviolet light, introduced into the cavity from a synchronized spark discharge in room air. Care was taken to attenuate the uv as needed to avoid lowering of the breakdown threshold. They found that even in the presence of triggering electrons from photo-electric emissions, the breakdown field

strengths for mixture of air plus 17.2 Torr of water vapor were up to about 25% greater than for dry air at the same total pressure, thus quantifying the effect of water vapor molecules in microwave plasma generation in their resonant cavity geometry.

In the case of breakdown fields required when using very short microwave pulses, MacDonald³⁰ also confirmed that “in the absence of an electron from some auxiliary source, the breakdown field measured would be very large, since the initial electrons would have to be provided by field emission or other surface effects.”

There are some distinct differences between the previous studies and our present work. First, our plasma chamber is not specifically designed as a resonating cavity for any specific mode; rather, two different glass flasks and two different microwave ovens of different sizes produced similar qualitative results. Second, no external source of triggering electrons either in the form of radioactive sources or synchronized photo-electric emissions were necessary to generate plasma in a microwave oven as seen in the video.³⁷ Third, theoretical calculations show that in a 700 W kitchen microwave (one of the microwaves that we used in our experiments) with a $0.34\text{ m} \times 0.44\text{ m}$ inner floor, the peak electric field of the microwave³⁸ is $E_0 = \sqrt{2I_{\text{ave}}/c\epsilon_0}$, where average intensity $I_{\text{ave}} = P/A$, $c = 3 \times 10^8\text{ m/s}$ is the speed of light, $\epsilon_0 = 8.85 \times 10^{-12}\text{ C}^2/\text{Nm}^2$ is the permittivity of free space, $P = 700\text{ W}$ is the power, and $A = 0.15\text{ m}^2$ is the area of the base. The calculated minimum electric field inside the flask in our microwave oven is thus $\sim 10\text{ V/cm}$ to spark plasma, in comparison to $\sim 10^2\text{ V/cm}$ or more in most of the previous studies for our operational minimum pressure, i.e., $\sim 100\text{ mTorr}$. Although some studies confirm the low electric field value inside an oven,³⁹ some numerical simulations using COMSOL⁴⁰ and passive measurements using a neon lamp⁴¹ show that the electric field inside a microwave oven can be much higher than the theoretically calculated value. A direct measurement of the electric field inside a microwave oven a few seconds after turning it on is thus needed to confirm the exact value of the minimum electric field needed in our experiments, and we invite the readers to design an experiment to do that.

II. PLASMA GENERATION BY A KITCHEN MICROWAVE

A. Materials and methods

The main component of this low-cost plasma etching system is a regular kitchen microwave oven and a vacuum flask/sample holder. Any robust, vacuum-gauge glass container will work, but we have found the Erlenmeyer flask is the simplest and least expensive. The flat bottom of this glassware gives extra stability during sample preparation which may be useful in a teaching environment. A valve must be attached to the flask stopper that can be opened while evacuating the chamber and closed again to seal and hold the vacuum. Speciality microwave-resistant valve parts can be purchased, but here we have sought a frugal alternative first. The ideal substitute for this part was determined to be the detachable two-part tips found on many burettes (Fig. 1). These parts consist of an all-PTFE housing and stopcock into which a glass tip is loosely inserted.

The glass tip of the burette can be easily removed by twisting it slightly. Next, a rubber stopper with a hole in the center,

such as those for inserting thermometers into reaction vessels, is used. This part is selected to fit the mouth of the Erlenmeyer flask described above. The glass burette tip above is then inserted through the bottom of the stopper until it protrudes from the top by about 10 mm. The PTFE stopcock housing is then placed back over the protruding burette tip. All of the edges of this system are then sealed with regular silicone glue. The seal is sufficient to hold a vacuum for many hours. We have also found that a properly matched microwave-safe PTFE/glass vacuum flow control adapters, such as StonyLab flow control adapters, attached to any glass container/flask, or modified Schlenk tube flasks with glass stopcock (GrowingLab), or similar stopcocks for glass vacuum desiccators can serve this purpose also (Fig. 2). The main point here is that the design of the container is not limited to the ones described here; rather, any microwavable vacuum container with a stopcock valve that can hold vacuum in the container can be converted into a plasma chamber. A vacuum hose can be directly attached to the top of the valve allowing the system to be evacuated to various pressures with a small vacuum pump. Once the properly evacuated flask is put in the microwave and the oven is turned on, after a few seconds glowing plasma forms inside the flask (please watch the video of plasma generation.³⁷) The time difference between the first observation of plasma and the time of turning on the microwave oven, i.e., the plasma initiation time, is recorded for different vacuum pressures. Furthermore, by flushing the evacuated system using a three-way flow control adapter with various gases like argon, nitrogen, oxygen, etc., this system can be used to determine the effect of different plasma chemistries. A schematic of the whole process is shown in Fig. 1.

In all of the following experiments, the substrate or sample is placed in an Erlenmeyer flask, the vacuum hose is attached, and the pressure is lowered with a vacuum pump to the desired value. The valve is then sealed and the flask transferred to the microwave oven. Once the microwave oven is turned on after a short delay the plasma sparks, and then the plasma is allowed to glow for the desired amount of time.

B. Characterization of the microwave-generated plasma

The plasma generated by this method is analyzed using two simple but revealing techniques: (a) recording plasma initiation time as a function of the vacuum pressure, which is also a measure of particle density at constant temperature, and (b) measurement of UV-visible emission spectra of the plasma glow. A Paschen-like graph^{42,43} is generated by plotting the plasma initiation time versus the vacuum pressure of the flask to varying level (Fig. 3). A trough-shaped curve resulted from this analysis, showing asymptotic tendencies at high and low vacuum pressures and a sweet-spot regime in between in which the plasma sparks within a matter of seconds. To generate a classical Paschen curve in a gas discharge tube, the breakdown voltage (or energy required for plasma initiation) between two electrodes is normally recorded as a function of pressure. However, in our system, the energy required for plasma generation is supplied through a constant power kitchen microwave, and the time the flask is exposed to microwave radiation determines the amount of energy supplied to the system. So, in our case we have plotted the microwave exposure time to initiate plasma as a function of the pressure of the flasks.

In our experiments, the microwave creates a sky-blue plasma in the flask that is visible during the first 1–2 s after

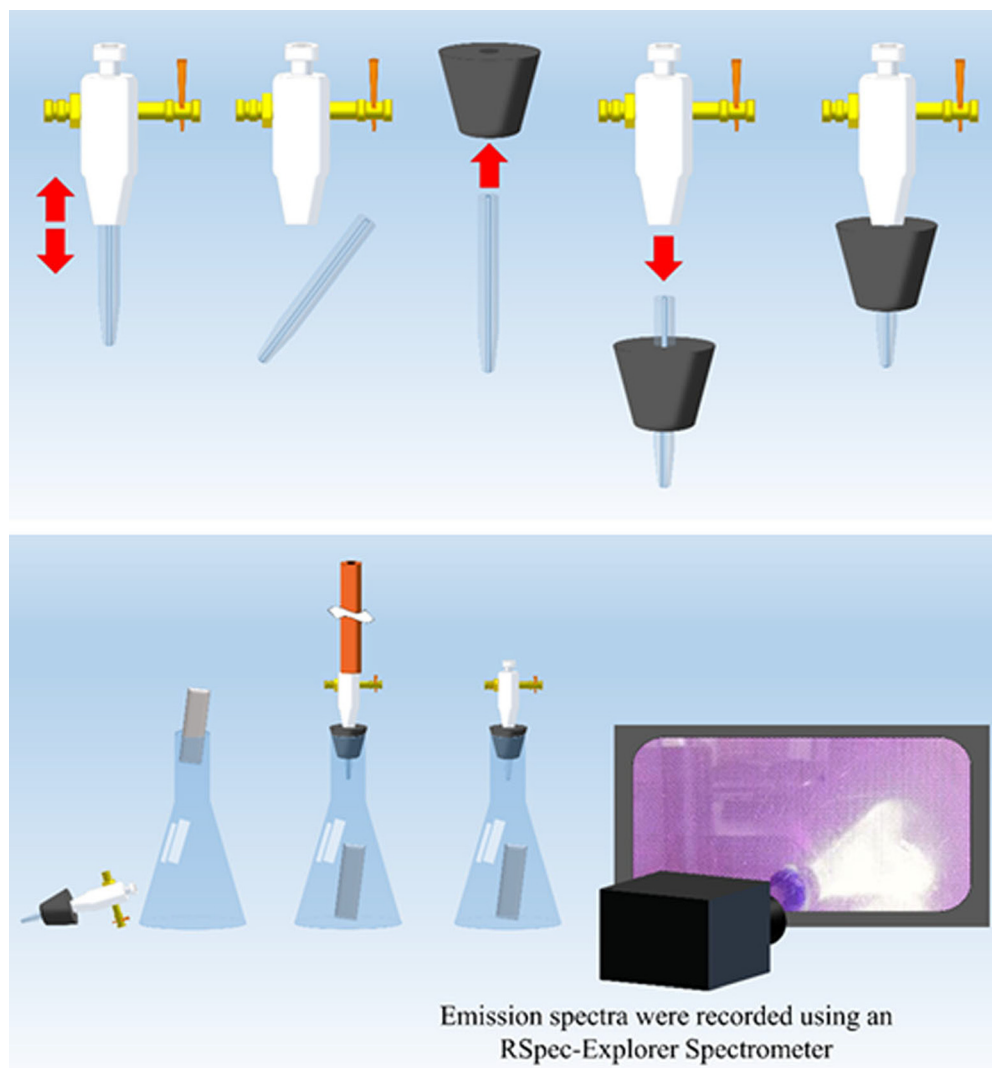


Fig. 1. A schematic of the construction of the sample holder for the microwave-generated plasma. One of the crucial parts needed is a simple air-tight valve. An inexpensive one can be fabricated from specialty parts as shown here, by modifying a PTFE and glass burette tip and rubber stopper. Bottom left to right: The sample is inserted into the Erlenmeyer flask; the vacuum valve is opened and the vacuum pump is used to evacuate the flask; the valve is closed and the vacuum hose removed; the flask is transferred to the microwave oven, and as the microwave is turned on, plasma is sparked to treat the surface for the desired length of time (Ref. 37). To characterize the spectrum of the plasma, this process is repeated without a sample while placing a UV-Vis spectrometer in front of the microwave window.

ignition (please see the figures in the supplementary material for illustrations). When the vacuum pressure is decreased further to 68 mTorr or less for a 250 ml flask and 69 mTorr or less for a 125 ml flask, the plasma ignition time tends towards infinity in this asymptotic limit. We have used two different kitchen microwaves with several different glass containers and the overall characteristic remains the same, although the plasma initiation times may vary slightly depending on the size and geometry of the microwave ovens.

UV-Visible absorption spectra of plasma are also recorded using a desktop CCD spectrometer (Exemplar, B&W Tek) and are shown in Fig. 4. The spectra of the microwave plasma clearly show the dominant nitrogen and oxygen peaks indicating ionization of these molecules. To understand and compare the microwave plasma generation with a DC discharge plasma, we also built a gas discharge tube with fixed electrodes, generated air plasma in it, and compared the spectra obtained from the gas discharge tube with that generated by the microwave. The reason behind this comparison is that gas discharge plasma is very well studied

and the plasma generation mechanism of a gas under a constant strong electric field leading to Townsend avalanche is well known.^{42,44} The striking similarity of both the spectra (Fig. 4) lead us to confirm that although the source to energize gas is different in two cases: (a) DC electric field in a gas discharge tube, while (b) electromagnetic waves in microwave-generated plasma, the chain reaction is somewhat similar, as described in the Appendix.

C. Classroom integration of this experiment/ demonstration

This simple plasma generation demonstration can be integrated into undergraduate physics classrooms and labs to teach several key concepts, such as the nature of electromagnetic waves, the kinetic energy of ions and electrons in electromagnetic waves, dipole moment vectors, force and torque generated by an electric field, etc. We have created several concept cartoon-based clicker questions on these key concepts, given in the Supplementary Material,⁶⁹ in order to



Fig. 2. An alternative microwavable vacuum container to generate microwave plasma.

explain this plasma generation process. We would encourage teachers and educators to use these concept cartoon-based clicker questions along with this paper and the featured article⁴⁵ to engage students in the classroom, initiate both student-instructor and peer-to-peer discussions, and go through brainstorming sessions.

III. APPLICATIONS OF MICROWAVE PLASMA

Plasma treatment is a well-known technique to remove any contaminant, change surface energy, and modify

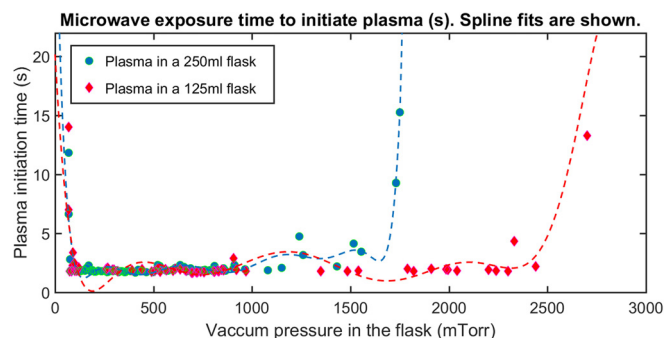


Fig. 3. Plasma initiation time measured and plotted as a function of vacuum pressure in two different flasks. A Paschen-like curve is observed when the pressure of the flasks are reduced and placed in the microwave oven to record plasma initiation time. At ambient pressures and high vacuum, the initiation time grows asymptotically, while in the intermediate regime, plasma sparks consistently within a few seconds. If the pressure in the flasks becomes too low, higher initiation time is observed pointing towards a low number density of molecules in the flasks and corresponding lower collision cross section. Data for two flasks, one 125 ml and the other 250 ml, show similar asymptotic behavior.

electrical and thermodynamic properties of a substrate for various purposes.⁴⁶ A change in the surface energy of a substrate can easily be ascertained by measuring the contact angle of a liquid on the surface. The contact angle of a liquid drop, i.e., the angle measured where a three-component interface exists between a liquid drop, a solid surface, and the ambient air, is measured by first taking cross-sectional pictures of the drop using a camera and then analyzing the contour of the drop using software. Normally, the designation of a hydrophobic material is reserved for surfaces with a liquid contact angle of greater than 90° . Contact angles of less than 90° indicate a hydrophilic material with good wetting properties.

A. PDMS surface modification by plasma treatment

A flat PDMS substrate is prepared by mixing the PDMS liquid elastomer and the curing agent (Sylgard 184) in 10:1 ratio followed by degassing and incubating for a couple of hours, resulting in a consistent, bubble-free soft solid block. Soft cured PDMS blocks are then cut to shape and placed in the vacuum flask. The flask is evacuated to 500 mTorr and placed in the microwave for plasma treatment. Once the plasma sparks, the PDMS blocks are treated for 2, 3, 4, and 5 s, respectively. The relative change in surface energy is evaluated by measuring the contact angle of a water microdroplet placed first on an untreated and then on the treated surfaces using a micropipette. We have used the open source image-processing software ImageJ with DropSnake plugin to measure the contact angle of the sessile micro-droplet.⁴⁷ Figure 5 clearly shows that the contact angle of water on the microwave-plasma treated PDMS sample increases nearly linearly with the treatment time indicating a direct relationship between the change in surface energy and microwave-plasma etching.

Surprisingly, this observation is exactly opposite the effect observed in PDMS surfaces exposed to RF-generated O_2 plasma⁴⁸ and also microwave-oven-generated O_2 plasma,⁴⁹ where exposure to oxygen plasma lowers the contact angle and makes the substrate more wetting. To further investigate what role microwaves play in this reversal of contact angle, we have investigated the effect of microwaves on a PDMS substrate with and without plasma (Fig. 5). It shows that while microwave (without plasma) has some small effect on making the PDMS surface less wetting as the contact angle rises from 63° to 70° with 10-s treatment time, the effect of microwave plasma is much larger as the contact angle changes from 63° to 159° with just 5 s of plasma treatment. We propose that the adsorbed water molecules on the surface of the PDMS substrate play a crucial role here. One plausible explanation is that the surface adsorbed thin film of water gets heated up by the microwave component and simultaneously a rapid interaction with high density ions through the plasma contacts embeds ions on the soft-baked PDMS substrate surface leading the substrate towards super-hydrophobicity. Also it is to be noted that the studies mentioned^{48,49} worked with a pure O_2 plasma, while in the current work the plasma seems to be heterogenous. This microwave-plasma treatment technique is thus useful for making hydrophobic surfaces for water-resistant coatings and other applications, which cannot be achieved through gas discharge plasma treatment, as well as for bonding PDMS to glass substrates and other applications, as investigated in detail in Sec. III D.

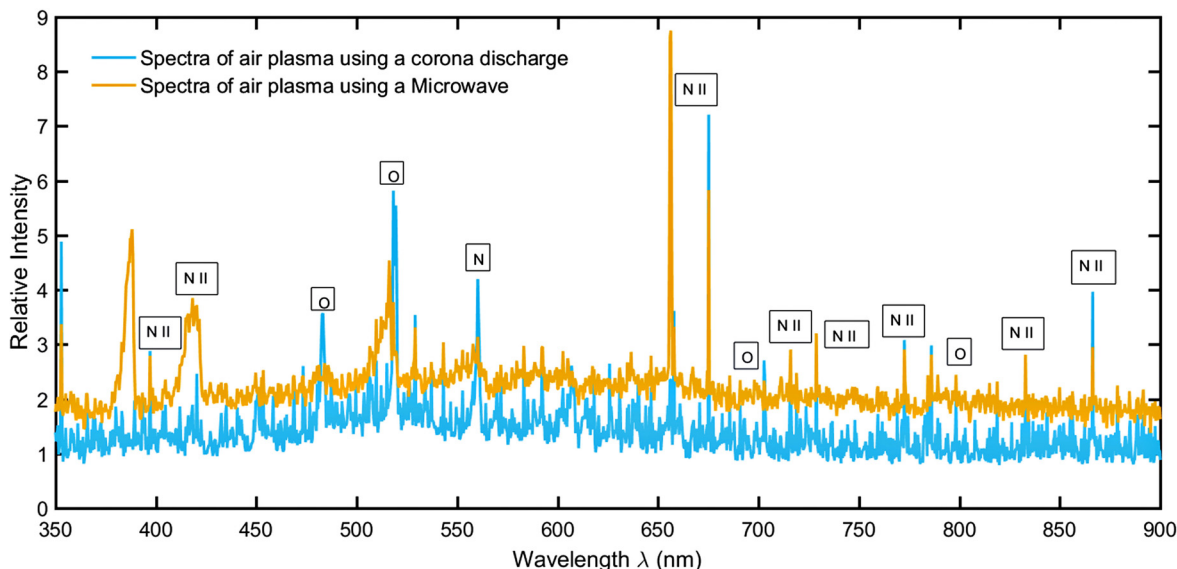


Fig. 4. The microwave-generated plasma spectra are compared with the corona discharge (gas discharge) tube air plasma spectra. Nitrogen and oxygen lines are visibly identified in both spectra.

B. Surface modification of ZnO thin films to change its opto-electrical properties

Plasma treatment of a metal oxide thin film can also change its opto-electrical properties. A film of ZnO particles was prepared as previously described.⁵⁰ Briefly, a suspension of ZnO particles were deposited on a glass microscope slide

from a 60% (v/v) solution of ethanol. The film was air dried for several minutes and then annealed on a hot plate by ramping the temperature from ambient to 500 °C in 7 min. Previous work has shown that as the ZnO film cooled, a layer of ambient moisture and gases, as well as ethanol which is trapped in the bulk microstructure of the ZnO, deposits onto the ZnO surface.⁵¹ This is verified by a strong OH band

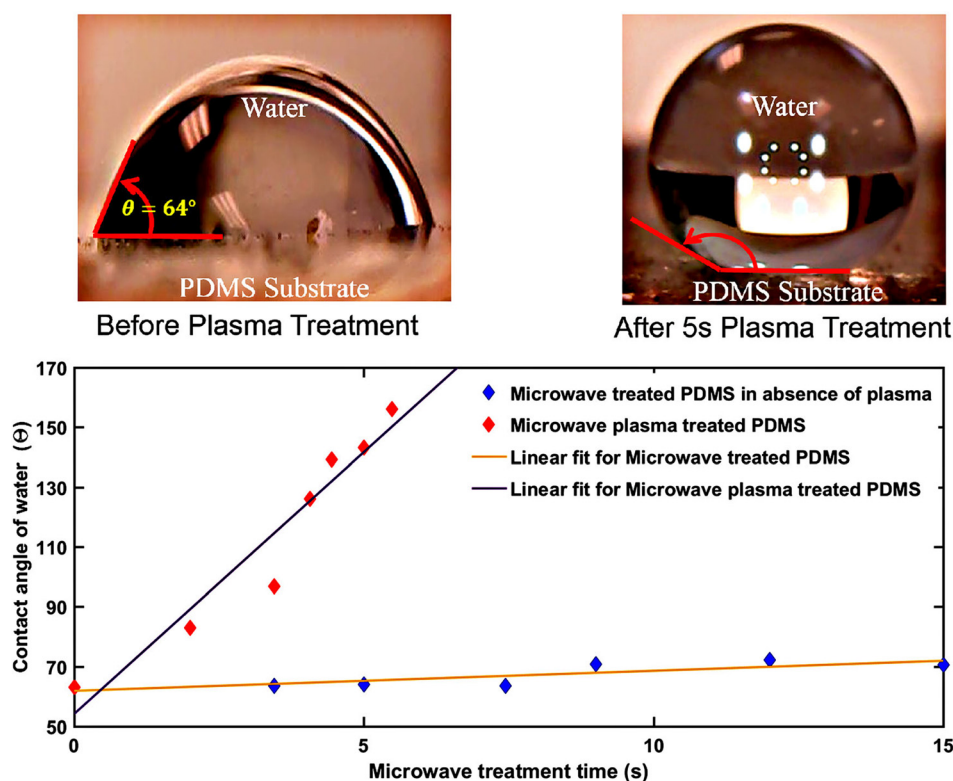


Fig. 5. Change in contact angle of water in a microwave oven, with and without plasma. Droplet pictures at the top show increasing contact angle with extended plasma treatment time by adding sufficient surface energy of the soft cured PDMS polymer substrate. To differentiate the effects of microwave and microwave-plus-plasma, one can choose a suitable pressure (say 2000 mTorr) and put the substrate in a 250 ml flask. As this pressure is above the higher asymptotic limit of the Paschen-like curve (Fig. 3) turning the microwave on won't spark plasma in the chamber. However, if we put an identical sample in a 125 ml flask at the same 2000 mTorr pressure and turn the microwave on, the substrate can be treated with both microwave and plasma, thus giving us a way to differentiate the effects of microwave plasma quantitatively.

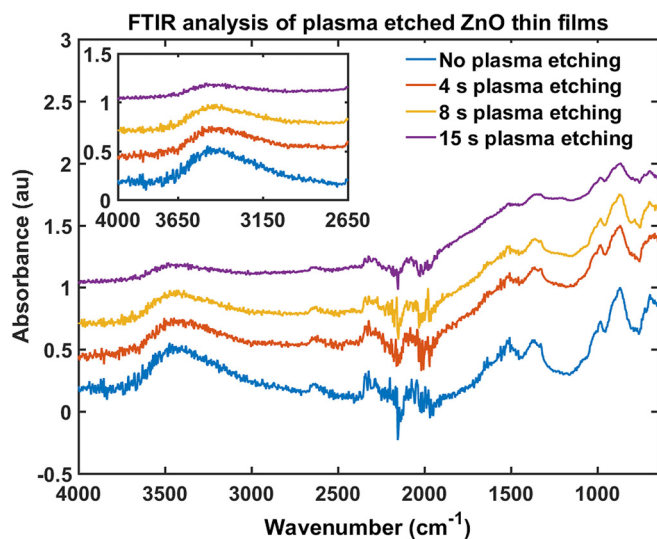


Fig. 6. Result of plasma etching a ZnO nanowire thin film. The film was fabricated as for all devices (deposition from ethanol suspension) followed by sintering. The etching was conducted for 4, 8, and 15 s. The inset shows the band at 3400 cm^{-1} due to the -OH bond in alcohols. The intensity of this signal clearly diminishes as the film is etched indicating a loss of electron-withdrawing groups from the surface of the material. The large signal at around 2100 cm^{-1} is from CO_2 , and the band at around 900 cm^{-1} is due to a small amount of glass particles that were scraped off the device substrate. The graphs shown above are the FTIR analysis of ZnO nanowire thin films; however other ZnO thin films made of ZnO particles or nanoparticles also show similar behavior under plasma etching.

centered at 3400 cm^{-1} in the FTIR spectrum of the film. The sample and substrate were transferred to the vacuum flask. The flask was then evacuated and the whole assembly was placed in the microwave oven as before. The plasma was sparked and allowed to interact with the ZnO surface for 4, 8, and 15 s. FTIR analysis of the treated surfaces in Fig. 6 shows a decrease in the overall intensity, especially of the OH band (centered at 3400 cm^{-1}). Our previous work⁵⁰ has shown that such modification of polycrystal and nanocrystal semiconducting films plays an important role in the electronic properties of the material.

C. Reduction of graphene oxide to graphene

Graphene is a 2D wonder material^{60–62} with many potential applications ranging from flexible electronics, energy storage, sensors to molecular sieving.^{63–66} One hurdle in further developing graphene applications commercially is the difficulty in preparation of bulk graphene in mass scale. One way of producing graphene is chemical vapor deposition (CVD), which is expensive and requires expensive instrumentation. Graphene is most easily produced in bulk by the reduction of graphite oxide solutions, but this can be time-consuming and is not an environmentally friendly process. Some recent works have shown alternative optical and thermal processes for obtaining reduced graphene oxide using a laser.^{67,68} Here we show that plasma generated by our simple system is capable of providing the local energy needed to reduce dielectric graphene oxide films to conductive graphene (Fig. 7).

First, graphene oxide (Graphenea Inc.) was drop casted onto polyethylene terephthalate (PET) film. After drying for 24 h at 50°C , the GO-coated foil was cut into squares. The sheet resistance of each square was measured using a

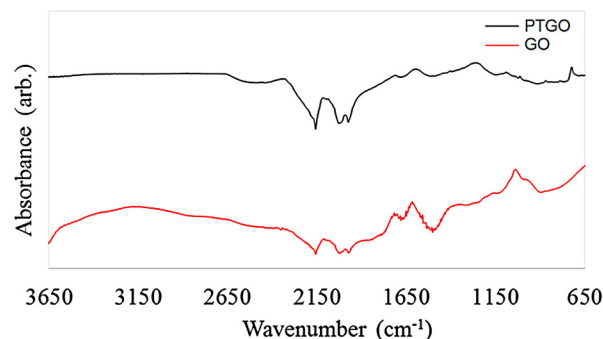


Fig. 7. Raman spectra and absorption studies for graphene oxide (GO) and plasma treated graphene oxide (PTGO) films.

Keithley Source Meter with a four-point Kelvin probe. The resistance prior to plasma etching showed a large value on the order of $10\text{ M}\Omega\text{ cm}^{-2}$ (Table I).

One graphene oxide square was taped on either end to a glass substrate and then placed in the plasma etching flask. It was then evacuated for 1 min (to a pressure of $\sim 300\text{ mTorr}$). The flask was placed in the microwave for 4 s. The other two squares were also treated for 4 s, but not affixed to substrates, and treated for 4 and 1 s intervals instead of continuous exposure. The three types are referred to as “supported,” “bursts,” and “free-standing,” respectively, as shown in Fig. 8.

The resistance following plasma etching was measured and showed a reduction of resistance of about two orders of magnitude. The lowest final resistance was obtained for the free-standing sample, but the heat from the plasma caused the plastic foil to warp, making this process less useful for flexible electronic applications.

D. Bonding of microfluidic channels to substrates

Many materials like polypropylene (PP), polyether ether ketone (PEEK), PDMS, or polyoxymethylene (POM) are extremely hard to bond with other materials. Requirement of high bonding strength, durable and irreversible bonding of metal, glass, and plastics present special challenges for the manufacturing industry. Plasma treatment of a surface with other applied cleaning procedures produces better adhesion capability and bonding strength on the surfaces to be joined in comparison to untreated substrates.

Microfluidics is an emerging technology with fluid flow in micrometer or smaller channels and has tremendous applicability.⁵² Applications for microfluidics include mobile chemical analysis,⁵³ medical diagnostics,⁵⁴ drug delivery,⁵⁵ soft robotics,⁵⁶ RNA encapsulation,⁵⁷ and nanomaterials synthesis⁵⁸ to name a few. In order to fabricate robust microfluidic devices, it is necessary to have a strong bond between the microfluidic polymer mold (typically PDMS) and the

Table I. Change of GO resistance before and after the microwave plasma treatment.

Substrate	Initial resistance $R_i\text{ (M}\Omega\text{ cm}^{-2}\text{)}$	Final resistance $R_f\text{ (M}\Omega\text{ cm}^{-2}\text{)}$	$\Delta R\text{ (%)}$
Supported	5.4	0.2	96.3
Free-standing	10.5	0.1	99.0
Bursts	7.9	0.1	98.7

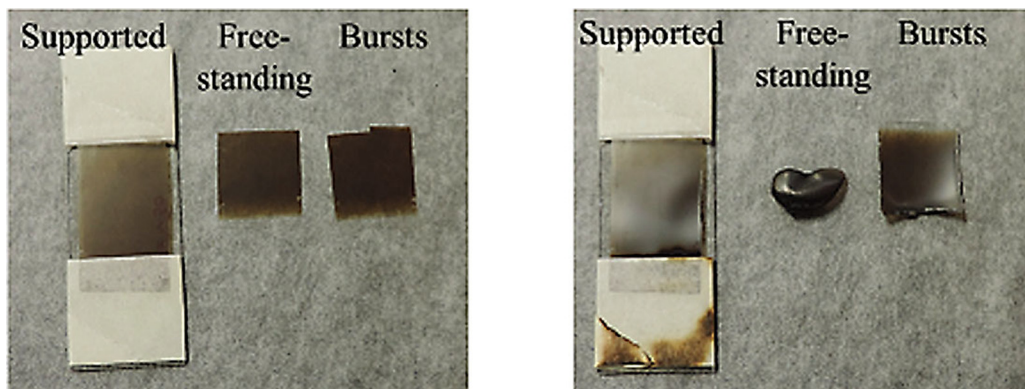


Fig. 8. Graphene oxide films before (left) and after (right) treatment in 4 s of air plasma. Clearly, the free-standing sample is not very useful for applications despite its large change in resistance. A similar change in resistance was attained by treating the bursts-sample with four separate 1-s pulses of plasma, which prevented the plastic substrate from warping significantly.

substrate. A strong bond prevents the device from leaking, becoming damaged, or developing passages by which the confined solutions could escape the network of channels. Plasma has been used in this application extensively, as it converts the exposed surface of the PDMS into dangling silane groups, allowing a very strong bond to glass to be formed. We demonstrate that the plasma generated in our simple microwave system can be used for this purpose.

The following steps can be taken to create PDMS microfluidic channels bonded on glass: (1) A microchannel mold is created (typically using photolithography or other methods). (2) A mixture of liquid PDMS and cross-linking agent is then mixed in a 10:1 ratio and poured into the mold in a petri dish. The petri dish is kept in an incubator at 65 °C for a couple of hours. (3) The hardened PDMS is taken off the mold. A replica of the microchannels is obtained on the PDMS block. (4) To complete the microfluidic chip and to allow the injection of fluids for future experiments, the inlets and outlets of the microfluidic device are punched with a biopsy puncher whose diameter is slightly less than the size

of the tubes to be connected. This will ensure tight fitting of the inlet/outlet tubes to the channels. (5) Finally, the side of the PDMS with open microchannels and the glass slide are treated with plasma to obtain closed channels with one flat inner wall of glass and all other walls of PDMS. (6) The plasma treatment irreversibly bonds PDMS with glass and makes the microfluidic chip.

A freshly made PDMS replica and a glass microscope slide in two different vacuum flasks were used in this experiment. The flasks were evacuated to 500 mTorr (0.5 Torr), and 3 s of plasma exposure was used on both of them. After plasma treatment, the glass slide was removed and placed on a flat surface. The PDMS sample was then removed and the treated surface is placed in the desired location on the glass slide; the two components were then pressed together gently and allowed to sit for ten minutes. A schematic of the whole process is shown in Fig. 9.

We have also measured the adhesive force of a PDMS surface before and after treatment using the force-distance microscopy mode of an atomic force microscope (Nanosurf

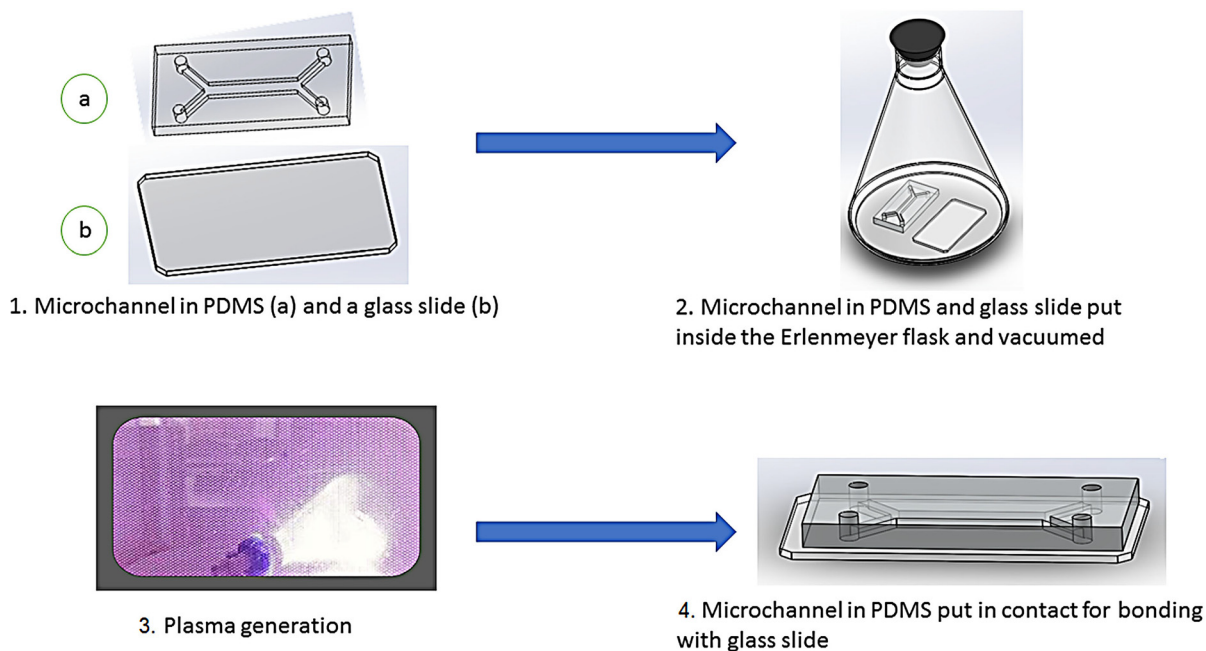


Fig. 9. A schematic to bond PDMS mold to glass to make microchannels.

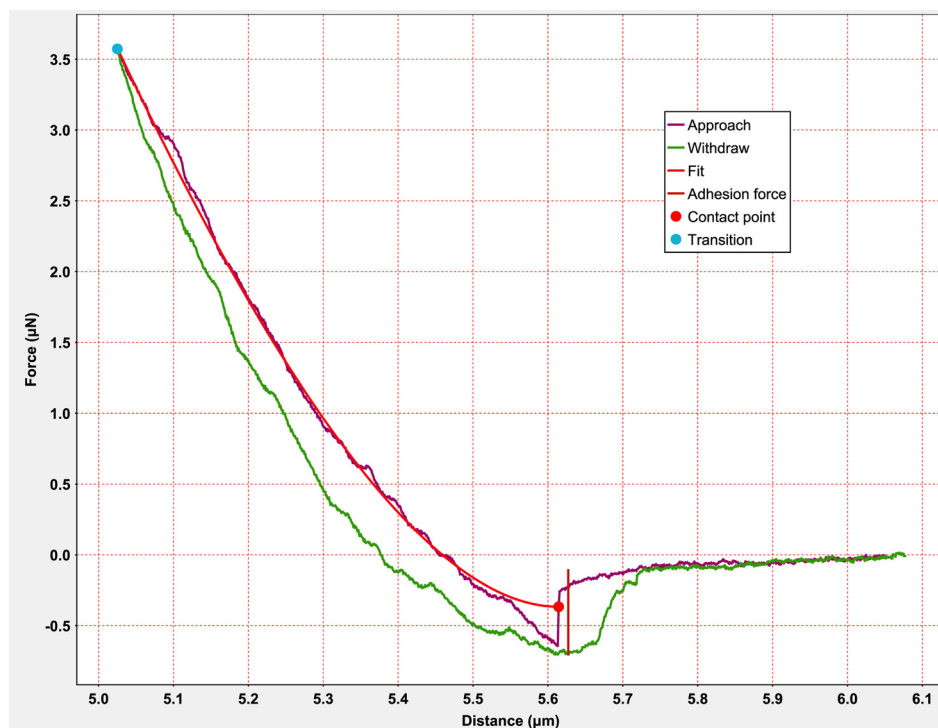


Fig. 10. Adhesive force of the untreated PDMS surface is measured using the AFM force spectroscopy method.

C3000 FlexAFM), and the representative graph is shown in Fig. 10. The relative change in adhesive force due to surface plasma treatment is shown in Fig. 11.

IV. CONCLUSION

In this work, we have demonstrated that a simple plasma generating device can be constructed from a household microwave oven and a vacuum flask. We have shown that this apparatus can be used in surface treatment of a substrate for several cutting-edge research applications. Varying degree of plasma exposure to a PDMS substrate leads to a change in surface energy, and hence a change in contact angle of a water droplet on the substrate was observed before and after the surface treatment. Upon exposure to plasma, a change in opto-electrical properties of a metal oxide semiconductor such as ZnO was demonstrated using FTIR spectra analysis. Significant change in electrical resistance of graphene oxide thin films was also observed as an effect of plasma exposure. Moreover, it was also shown that irreversible bonding between certain elastomers like PDMS and

glass can be achieved to make microfluidic channels with this simple technique. Needless to say that the domain of these applications is enormous and our hope is that this paper, through classroom/lab integration and this frugal alternative to conventional and expensive plasma treatment, would enable more researchers to delve into these cutting edge research areas leading to more innovation and discovery.

ACKNOWLEDGMENTS

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APPENDIX: INTEGRATING THIS RESEARCH TO TECH BASIC CONCEPTS OF EM

The plasma generation experiment using a kitchen microwave oven can be used as a classroom demonstration in an effort to engage students in discussions of some important concepts in electromagnetism in undergraduate physics. We have created a pool of multiple choice concept cartoon questions to explain our microwave generated plasma formation process and to initiate discussions on the key concepts of EM, such as the nature of electromagnetic radiation, the

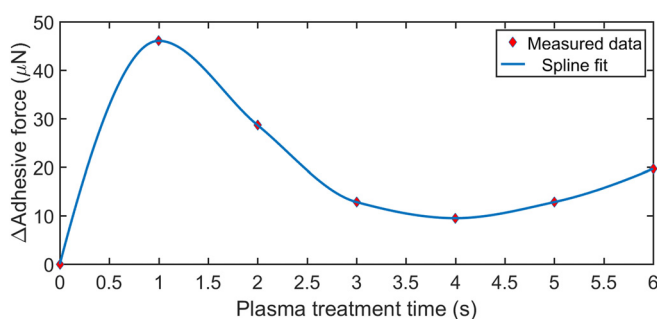
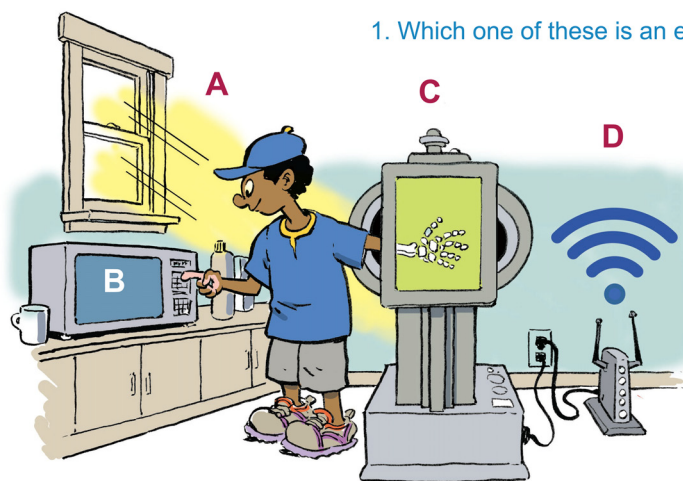


Fig. 11. Adhesive force of the PDMS surface is measured using the AFM force spectroscopy method before and after various degrees of plasma treatment.



1. Which one of these is an electromagnetic wave?

- A. Sunlight
- B. Microwaves
- C. X-rays
- D. Wi-fi signal
- E. All of the above



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Fig. 12. This slide elaborates that we are surrounded by electromagnetic waves. Other slides are available in the Supplementary Material (Ref. 69).

relationship between force/kinetic energy of a charge and the applied electrical field, dipole moment vector, and the torque applied by an oscillating electric field, collision cross section, cascading effect to generate a plasma, etc. Impact of similar cartoon questions in the classroom were measured and reported.⁵⁹ The first slide is shown here in Fig. 12 as an example, and all nineteen are in the online Supplementary Material.⁶⁹ High resolution pictures of these slides can also be obtained by emailing the corresponding author of this paper.

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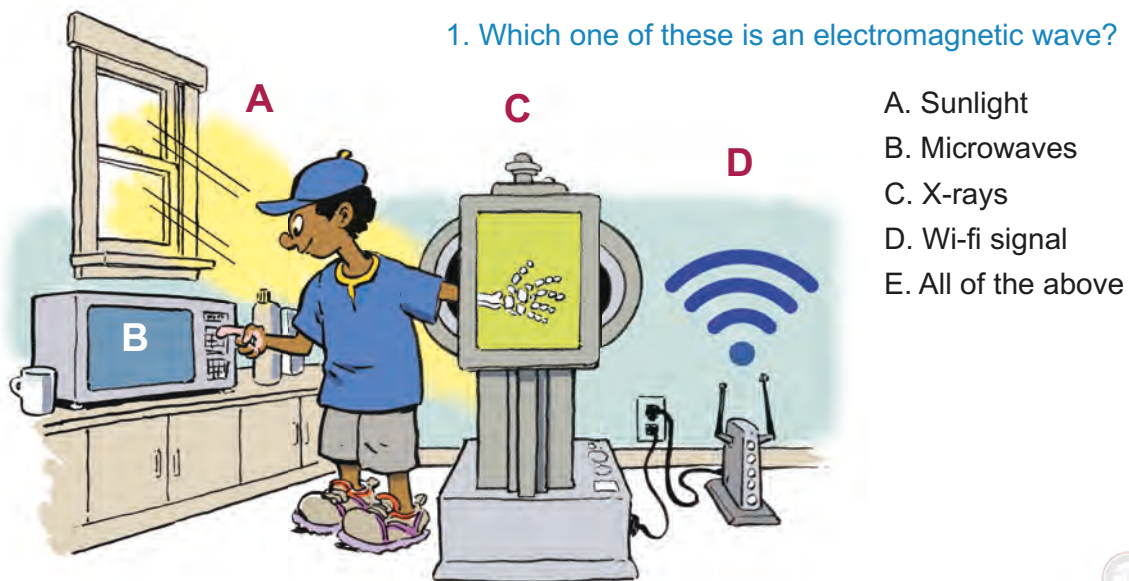
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- ⁶⁹See Supplementary Material at [supplementary material](#) for Cartoon Clicker questions.

Supplementary Information: Plasma Generation by Household Microwave Oven for Surface Modification and Other Emerging Applications

American Journal of Physics 89, 372 (2021); <https://doi.org/10.1119/10.0002706>

CLASSROOM SLIDES

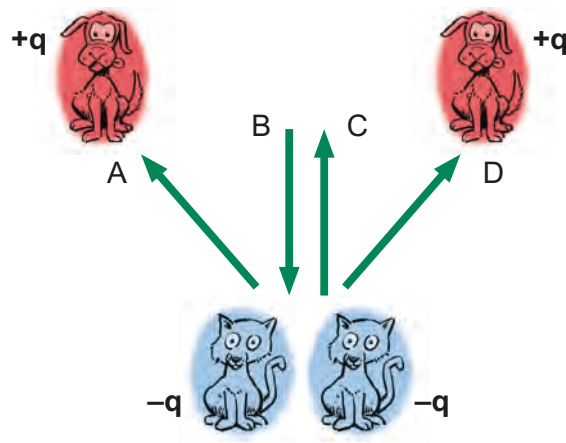


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FIG. 1. This slide elaborates that we are surrounded by the electromagnetic waves.

2. Two positive and two negative charges form dipoles as shown below.
Which arrow shows the resultant electric dipole moment vector?

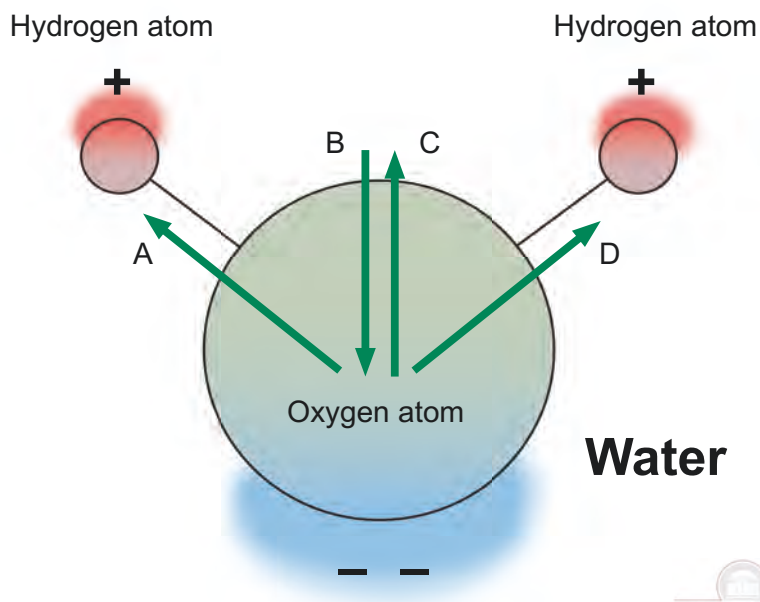


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FIG. 2. This slide can be used to elaborate the concept of dipole moment vector.

3. A **water molecule, H_2O** , is a dipole. Electrons hug the oxygen atom, so its end of the molecule carries a slight negative charge. The end with two hydrogen atoms has a corresponding positive charge.

Which arrow shows the direction of the water molecule's dipole moment vector?

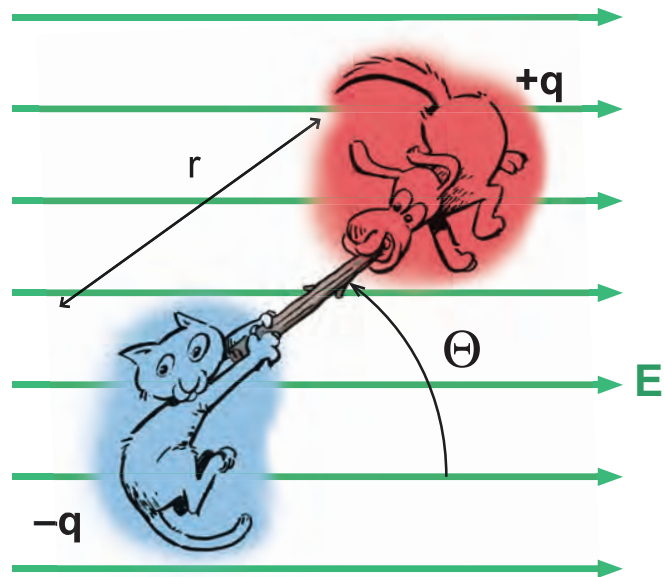


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FIG. 3. This slide shows the direction of dipole moment of water molecules.

4. In a uniform electric field $\mathbf{E}$, a dipole will rotate. If the dipole's charges are $\pm q$ and its length is r , what **torque** does the field generate on the dipole?

- A. $qEr \sin \theta$
- B. $qEr \cos \theta$
- C. $qEr \tan \theta$
- D. $qEr \cot \theta$

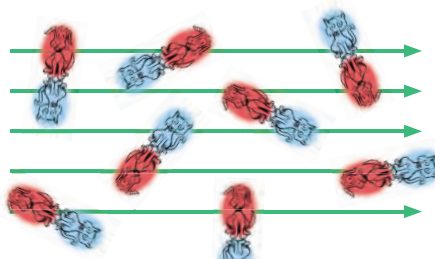


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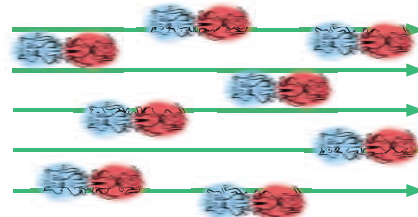
FIG. 4. This slide leads to the discussion of torque applied by an electrical field on a dipole.

5. A bunch of tiny dipoles are flying around in random directions and orientations. What happens when a **uniform electric field** is turned on?

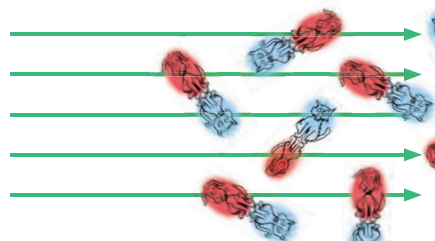
A. Nothing.



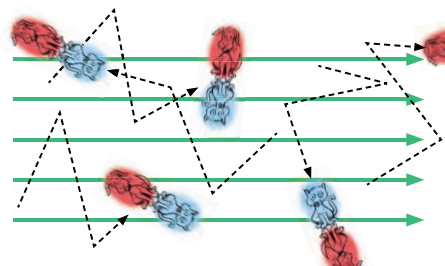
B. Torque on the dipoles aligns them along the field lines.



C. The dipoles begin moving along the field lines.



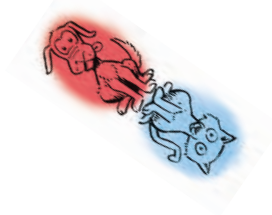
D. The dipoles begin to zig-zag.



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FIG. 5. This slide shows how a group of dipoles align along the direction of a steady electrical field.

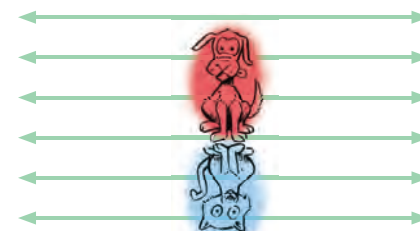
6. What happens to a dipole in an **oscillating** electric field?



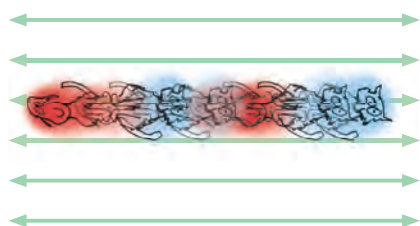
A. Nothing.



B. It aligns perpendicular to the field lines.



C. It slides back and forth along the field lines.



D. It spins or wobbles in sync with the field oscillation.

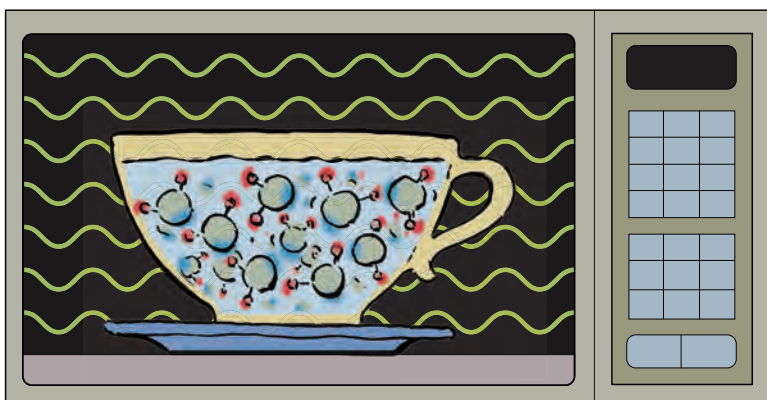


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FIG. 6. This slide elaborates oscillation of a dipole in an oscillating electric field. It may lead to a discussion of behavior of polar molecules like water in an oscillating electric field like microwave.

7. A cup of water's temperature goes up in a microwave oven. Why?



- A. There is hidden fire in microwaves.
- B. An oscillating electric field spins/wobbles water molecule dipoles, which agitate each other by intermolecular forces.
- C. Microwave particles move water molecules by bouncing off them.
- D. Microwaves heat the air inside the oven, which warms the water by convection.

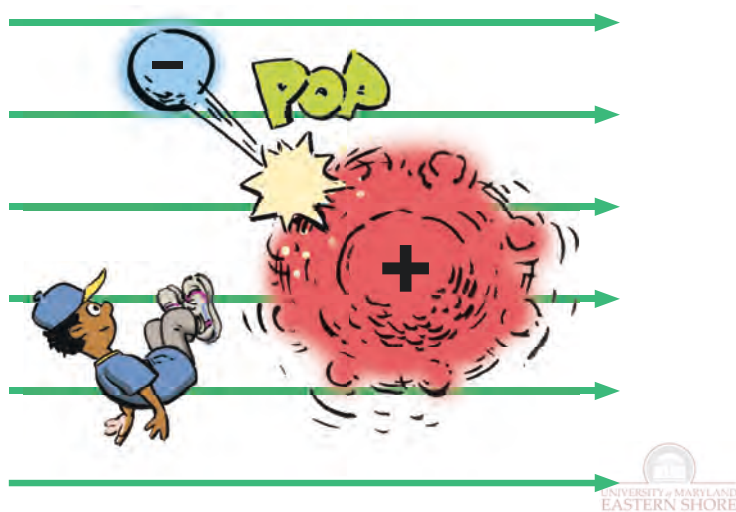


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FIG. 7. This slide illustrates that in a kitchen microwave oven water molecules oscillate in an oscillating electric field. However, due to hydrogen bonding between the molecules and viscosity of water the oscillating molecules rapidly dissipate energy, in turn heating up the substrate. It may lead to a discussion of microwave heating mechanism.

8. Powered by microwaves, a water molecule spins so fast that it throws off an electron. What happens to the negatively charged electron and the now-positively charged water ion?

- A. Nothing.
- B. The electron will return to the ion to make a neutral water molecule.
- C. The positive ion will accelerate in the direction of the electric field, and the electron accelerates in the opposite direction.
- D. The electron will accelerate in the direction of the electric field, and the positive ion accelerates in the opposite direction.

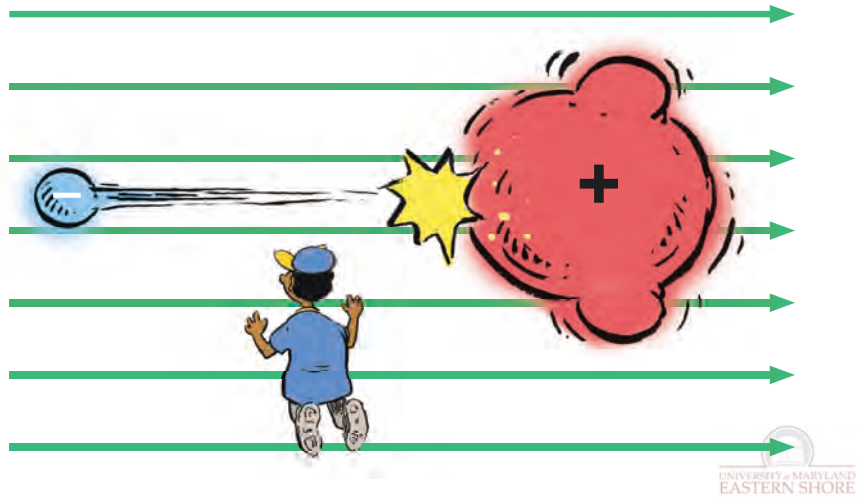


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FIG. 8. This slide discusses about rotational kinetic energy of polar molecules in an oscillatory electric field. Suppose inside a microwave oven there is a container filled with air and some stray water vapor molecules are in there. Water is a polar molecule with dipole moment p ($\sim 6.2 \times 10^{-30}$ C.m) [1],[2] and it is shown in Fig. 6 that oscillating electric fields can generate vigorous rotation/oscillation in polar molecules. An order of magnitude calculation of this torque τ ($\tau = \mathbf{p} \times \mathbf{E}$) produced by a household microwave with frequency[3] $f = 2.45$ GHz and calculated amplitude of electric field $E \sim 2 \times 10^3$ V/m, shows that the microwave electric field may generate an enormous angular acceleration α of a water molecule ($\alpha = \tau/I \sim 10^{20}$ rad/s²), $I \sim 3 \times 10^{-47}$ Kg.m² being the moment of inertia[4] of water molecule about the specific rotation axis. Moreover, we know that in practical cases electrical field inside a kitchen microwave oven can be orders of magnitude more[5] and if that is the case, then the rotational energy of the stray water vapor molecules would be even more. This scenario is one possible route to generate primary electrons. However, more work is needed to confirm this scenario.

9. When an electron breaks away from the molecule, an electric field will move them in opposite directions. Which statement is true about the magnitude of their accelerations, given that a water molecule is about 33,000 times as massive as an electron?

- A. Electron and water ion accelerate equally.
- B. The electron's acceleration is greater than the ion's.
- C. The ion's acceleration is greater than the electron's.
- D. Both particles decelerate.

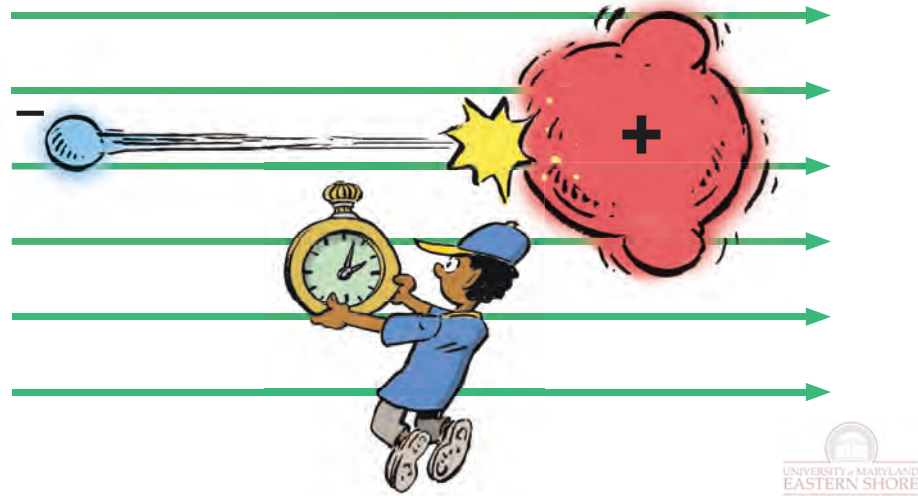


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FIG. 9. This slide reiterates the effect of instantaneous electric field on positive and negatively charged particles.

10. An electron flies off a (much more massive) spinning water molecule. In an electric field, both particles accelerate. At **t seconds' time** after the breakaway, which particle has greater **kinetic energy**?

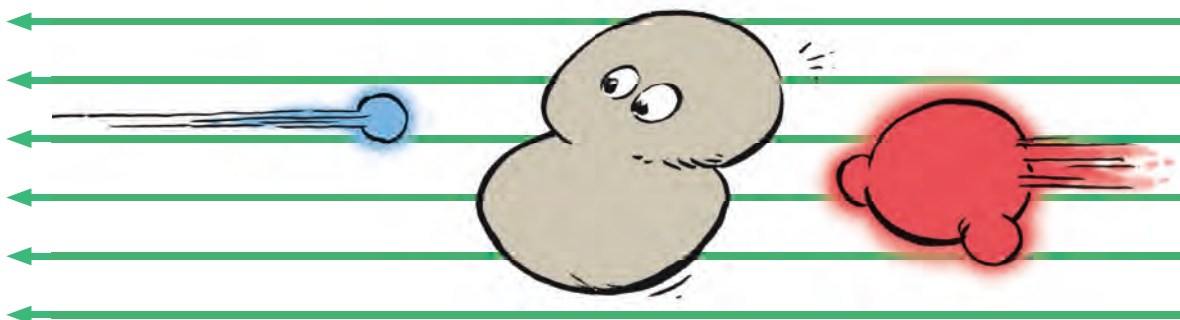
- A. The electron.
- B. The water ion.
- C. They have equal kinetic energies.



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FIG. 10. This slide can be used to initiate discussions on the effect of mass of the charged particles under the influence of an electric field on their kinetic energies after equal time interval. In case an electron of mass m_e and charge $|q_e|$ is generated by collision along with a massive ion of mass M_i and charge $|q_e|$ and both of them accelerate in an average electric field E , the ratio of kinetic energies of an electron and the ion can be obtained as $K.E_e/K.E_{ion} = M_i/m_e$. As $M_i/m_e \gg 1$, it is clear that electrons would be much more energetic in comparison to the massive ions in ionizing the gas in the flask. It is also well known[6] that the ion-molecule collision frequency is about $\sqrt{(m_e/M_i)} \ll 1$

11. You're a gas molecule in the chamber of a microwave oven. Electrons are flying off of water molecules. Which kind of particle can do you more damage, electrons or ionized molecules?



- A. Electrons, because they have higher kinetic energy.
- B. Ions, because they are more massive.

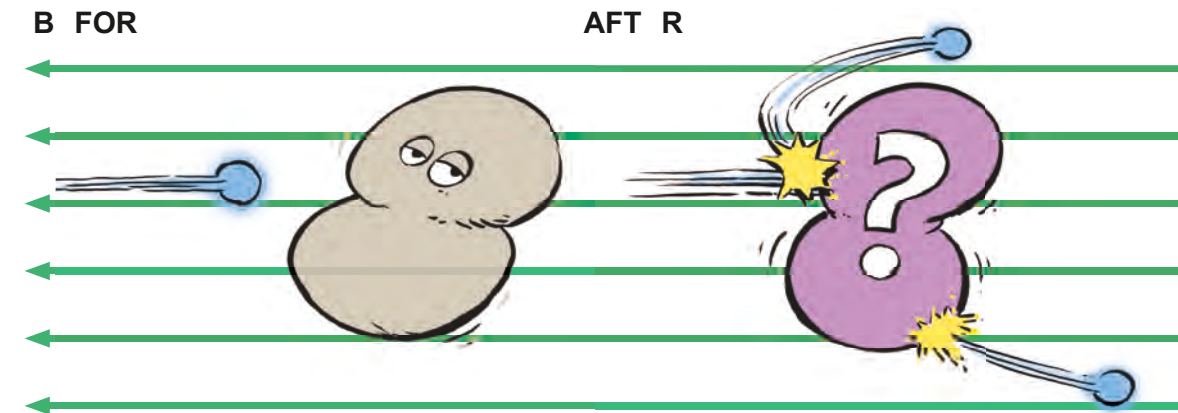
- C. Both are equally likely to do damage.
- D. Why worry? How dangerous is a tiny particle, anyway?



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FIG. 11. This slide is a follow up question on the effect of the mass of the charged particles on their kinetic energies when they are under the influence of an electric field.

12. If a high-energy electron hits a neutral molecule, bounces off, and knocks off another electron from the atom in the process, what happens to the atom's charge?

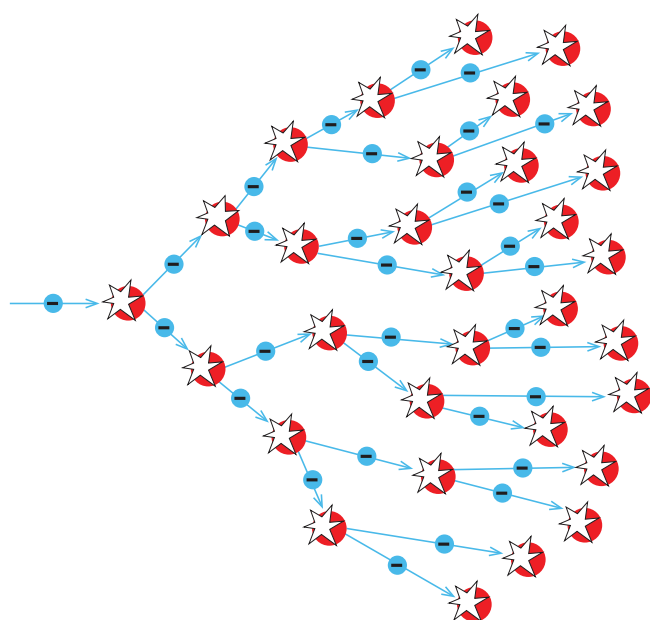


- A. It becomes negative. B. It becomes positive. C. It remains neutral.



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FIG. 12. This slide demonstrates the mechanism of secondary electron generation and ionization of an atom/molecule by the primary electron.



13. A sealed flask containing water vapor and other gases is put into a microwave oven. When the oven is turned on, what happens to the number of **free electrons** and **ionized molecules** in the flask?

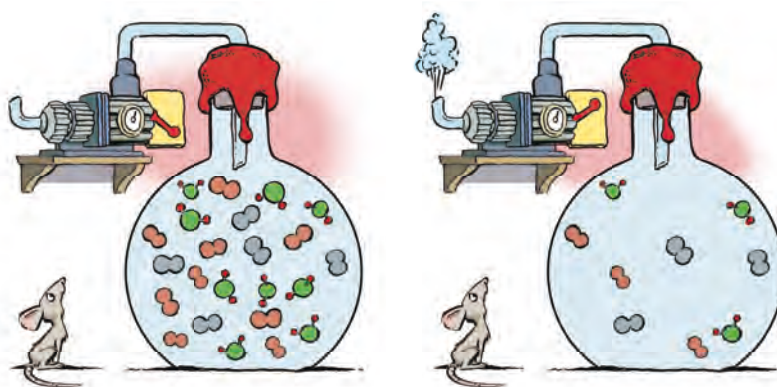
- A. The number of free electrons rises, while the number of ionized molecules falls.
- B. The number of free electrons remains constant, while the number of ionized molecules rises.
- C. The number of free electrons and ionized molecules both rise rapidly.
- D. The number of free electrons and ionized molecules both fall.



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FIG. 13. This slide demonstrates the chain reaction and the path towards plasma generation by Townsend type avalanche effect.

14. The liberated electrons' **mean free path** is the average distance they fly before colliding with something. If gas pressure is reduced by pumping gas out of the flask (at constant temperature), what happens to the electrons' mean free path?



- A. The mean free path decreases (electrons don't fly as far) because molecules expand to fill the space.
- B. The mean free path increases (electrons fly farther) because there is more space between molecules.
- C. Mean free path does not change because the electric field in the microwave oven is oscillating.

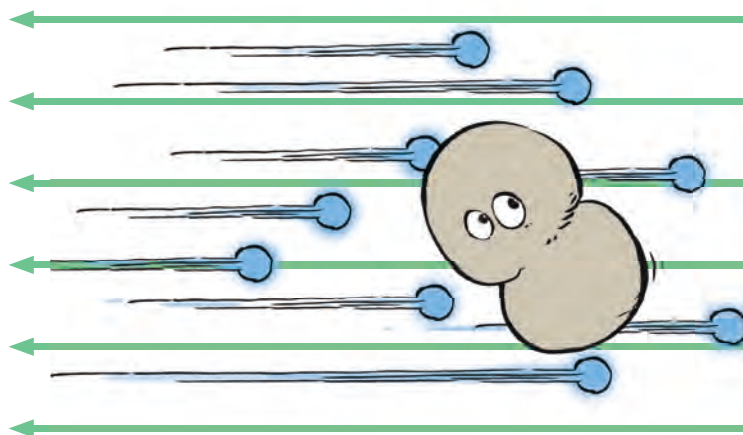


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FIG. 14. This slide demonstrates the effect of gas pressure on the mean free path. Less pressure means less density and more gap between the gas particles.

15. If electrons' mean free path **increases** in an electric field, what happens to the electrons' **average kinetic energy**?

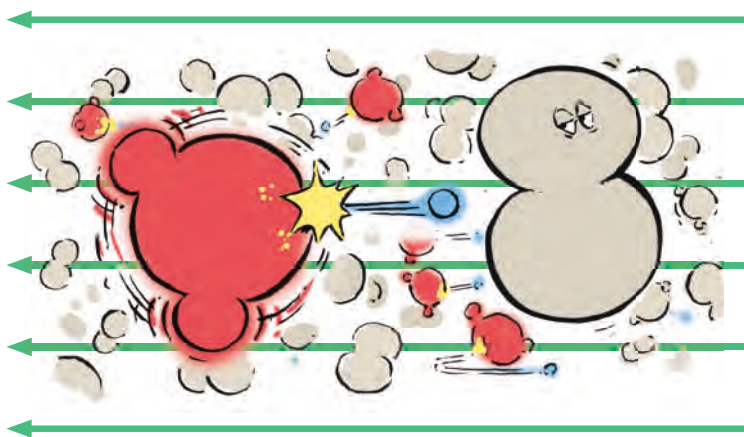
- A. The kinetic energy of electrons does not change in an electric field.
- B. Average kinetic energy goes down because the particles decelerate over time.
- C. Average kinetic energy goes up because the particles have more time to accelerate before colliding with something.
- D. There is not enough information to draw a conclusion.



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FIG. 15. This slide demonstrates that longer mean free path means more time for the electrons to gain more kinetic energy before the next collision.

16. When gas density is very high, and electrons' mean free path is very short, how is plasma formation affected?



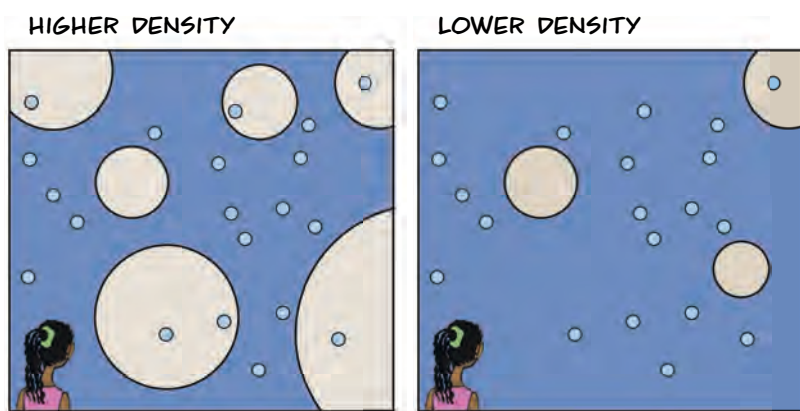
- A. Plasma will form more rapidly because electrons will quickly collide with molecules.
- B. Plasma will form more slowly (or not at all) because electrons will not pick up enough energy to ionize molecules.
- C. Gas density has no effect on plasma formation.



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FIG. 16. This slide demonstrates that shorter mean free path means less kinetic energy acquired by the electrons before the next collisions.

17. Bianca is watching a lot of electrons flying in the same direction away from her. The **collision probability** between the electrons and the gas molecules is (roughly speaking) the amount of Bianca's field of view that is taken up by molecules. What happens to collision probability as **gas density changes**?



(IMAGINE THAT PALE BLUE ELECTRONS ARE FLYING INTO THE SCREEN.)

- A. Collision cross section does not depend on gas density.
- B. Collision cross section goes down with increased density.
- C. Collision cross section goes up with increased density.

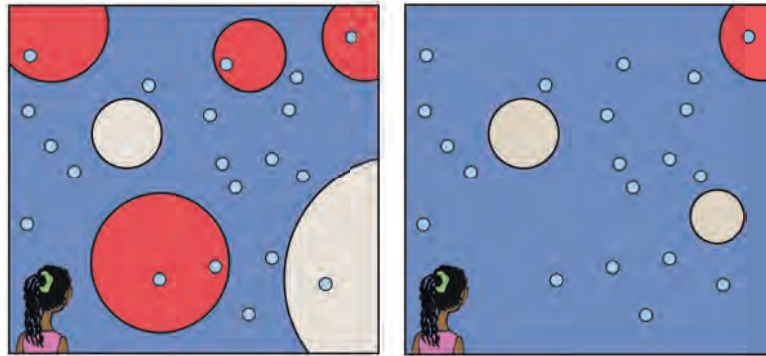


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FIG. 17. This slide demonstrates that effect of pressure on the probability of collisions of electrons with other constituent gas molecules. Average collision cross section depends on the gas molecule density (or pressure) in a secondary way, in so far as the electron energy depends on the gas density (and mean free path).

18. How does the probability of collision between the electrons and the gas molecules affect plasma formation?

(IMAGINE THAT
PALE BLUE
ELECTRONS ARE
FLYING INTO
THE SCREEN.)

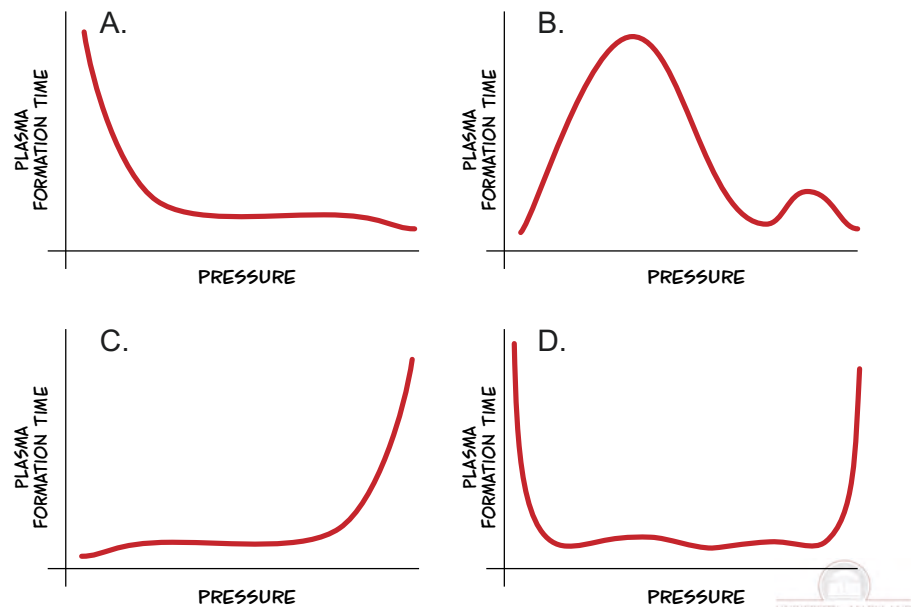


- A. When collision cross section is lower, ionizations are less probable, and plasma formation will be slower.
- B. When collision cross section is higher, ionizations are more probable, and plasma formation will be faster, provided that electrons are energetic enough.
- C. Both A and B are true.
- D. There is not enough information to draw a conclusion.



FIG. 18. This slide initiates a discussion on how decreasing gas pressure may decrease collision cross section and in turn reduce the probability of generating higher order electrons in turn adversely affecting the possibility of plasma generation.

19. We have seen that at constant temperature, **very low** gas pressure reduces **collision probability**, while very **high** gas pressure reduces electrons' **average kinetic energy** to knock out electrons and ionize gas molecules. Which of these graphs best represents plasma formation time as a function of pressure?



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FIG. 19. This slide initiates a discussion on the asymptotic nature of plasma initiation with respect to the pressure of the gas. We have seen that at constant temperature, very low gas pressure reduces collision cross section, while high gas pressure reduces electrons' average kinetic energy in between two successive collisions. Thus there should be a sweet spot between these two competing parameters where the condition for plasma generation is just right.

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