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The Domestic Microwave Oven as a Rapid Prototyping Tool

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Abstract

This paper reviews the use of the domestic microwave oven as a rapid processing multi-task prototyping tool. The investigation includes the use of these ovens for both plasma and non-plasma treatments, for the use in materials processing. The use of non-plasma microwave ovens for the processing of organic compounds with, or with, susceptors, ultraviolet irradiation and ultrasound irradiation is reviewed. By modifying the ovens, they can be used for plasma treatments, an example being the plasma sealing of PDMS for use in the fabrication of microfluidic devices and the plasma syntheses of carbon nanomaterials. The examples outline the potential as well as the challenges of the microwave irradiation for material processing, particularly when combined with UV/Vis as well as ultrasonic treatments.

***Keywords:* Microwave oven; Plasma; Organic compounds; Susceptors; Photochemistry; PDMS; Carbon nanostructures**

Introduction

For the past seventy years, domestic table top microwave ovens, operating at a frequency of 2.45 GHz, have been widely used for both defrosting and cooking food [1,2]. Increasingly microwave irradiation is also used as an energy source for the synthesis of organic and inorganic materials [3]. Given the ovens typically cost 50 to 60 Euros for a basic unit (cost in 2018) they are ideal system for conversion into plasma reactors as used in plasma-enhanced microwave processing of engineering materials [4,5], poly(dimethylsiloxane) (PDMS) [6] and carbon nanostructures [7]. Law and Denis published details on the conversion of microwave ovens into plasma reactors and their calibration [8], as well as for the plasma processing of organic compounds and biomaterials [9]. The most common feature in these ovens/plasma reactors is their packaged cavity-magnetron [10,11] (operating at a free running frequency of fo = 2.45 ± 0.1 GHz λ_o ~ 12.2 cm) [12] that is connected to a multimode resonant cavity (MRC) via a matched length of transverse electric (TE10) waveguide. Using this configuration, the MRC is loaded with the material that is to be processed resulting in a change in the Q-factor of the MRC from unloaded to load. As the magnetron is free running, typically 200 MHz within its -3dB bandwidth, impedance changes in the mono-mode waveguide, due to non-coherent microwave energy reflected back, result in frequency pushing of the magnetron. Thus, the microwave oven achieves a degree of auto-impedance matching with no other impedance matching apparatus required.

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The domestic microwave oven is used to cook a wide range of foodstuffs with different portion sizes, along with other non-food products: for example, it has been used for the thawing of blood plasma [13] the cleaning of dentures [14] and sterilization of microwave proof plastic food containers [15,16].

For the use of the domestic microwave oven in laboratory based chemical engineering experiments, a key consideration is the use of either batch or flow-through processing, as the outcome will influence scaling-up to full production. In 2013, Lo [17] outlined that building the Lab On a Chip (LOC) or Micro Total Analysis Systems (μTAS) strategy employed in PDMS-based microfluidic devices provides a scale-out capability (adding more components in parallel to spread risk) rather than conventional scale-up production that is more expensive and carries more uncertainties. Therefore, this paper looks at scale-out using the relative simple batch process rather than the flow-through process that requires a detailed knowledge of chemical residence time within the reactor. It is also worth noting that commercial mono-mode fixed waveguide microwave systems designed to treat fixed volumes of materials [9] at a relative high cost may not be able to handle the multi-task requirement at the prototyping stage.

When using a domestic microwave oven outside its original design specification, additional safety issues must be considered. These are: monitoring microwave leakage at the door and surrounding oven surfaces (the maximum power permitted is 5 $mW.cm^{-2}$ at the outer surface of the oven), the handling of hot materials including glass reaction vessels, handling of superheated fluids, low pressure vacuum systems and plasma UV radiation.

The aim of this paper is to review the domestic microwave oven as a rapid plasma-processing multi-task prototyping tool. With this aim in mind, section 2 provides a reference baseline on non-plasma microwave processing. The baseline includes syntheses of organic compounds (section 2.1) as microwave susceptor that convert microwave energy in to heat (section 2.2), microwave photochemistry (2.3) and microwave treatment of water (2.4) and ultrasound assisted microwave processing (2.5). Section 3 examines the microwave oven as a teaching resource in an education environment and at home. Section 4 exemplifies the multi-task of the microwave oven using two contrasting plasma process tasks – that of the reversible and irreversible sealing of PDMS as used in fluidic devices, as well as the manufacture of carbon nanostructures. Section 5 provides a discussion and future work. The paper reviews microwave and plasma microwave ovens and, accordingly different units of power and vacuum pressure are reported and converted to the equivalent SI unit of pressure (Pascal).

Non-plasma processing within the microwave oven

Rapid synthesis of organic compounds in microwave ovens: Once low-cost domestic microwave ovens become widely available in the 1970s, chemists started to report on preliminary work using the oven for synthesis of organic compounds (esters from carboxylic acids, carboxylic acids from alkyl benzenes and amides [18]. Their result indicated that reaction rates carried out in sealed 120 to 300 ml berghof and Teflon containers, could be up to 1240 times more rapid compared with classical reflux methods. Gedye et al went on to expand the preliminary work (and set out safety procedure for handling microwave heated organic compounds) within two full-length papers: one in 1988 [19] and the second in 1991 [20]. In these two papers, the microwave oven used was the Toshiba ER-800 BTC set to a power level of 180 to 560 W, with typical process times between 1 and 5 minutes. By 2001 specific system and mechanism properties different to conventional heating became apparent [21]. Four of these properties are as follows:

1. For liquids and materials contained within a vessel, conventional heating is performed by conduction and convection from the vessel wall to the center of the material so forming a thermal gradient, which drives the heat through the containing vessel wall and into the material medium (**FIG. 1a**). In contrast in a microwave oven the heating mechanism is through exposure to the electromagnetic waves. For example, polar liquids (i.e. many solvents) are very efficient in microwave energy absorption due to the interactive rotation of the polar liquid dipoles causing molecular agitation and intermolecular friction generating heat within the liquid and so raising the balk liquid temperature more rapidly compared conventional heating, (**FIG. 1b**).

2. The merits of using mono-mode microwave waveguide systems or multimode microwave oven is an issue when comparing the synthesis of organic compounds. For example superheating effects are readily observed in multimode microwave ovens in the absence of any stirring.

3. When the microwave energy is turned off conventional thermal chemistry continues.

4. In the reaction between selected solvents and aromatic hydrocarbons, including C70 fullerene, the microwave irradiation power level alters the ratio of isomer products.

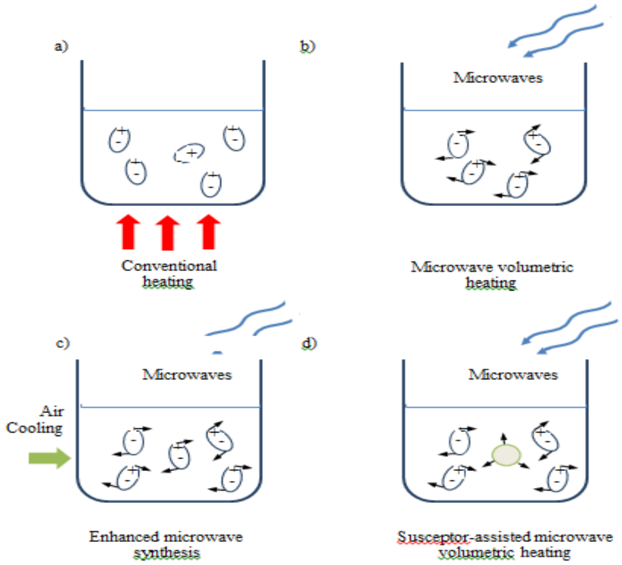

FIG. 1. **a) Schematic showing four methods for processes polar solutions: conventional heating, b) microwave heating, c) enhanced microwave synthesis, d) susceptor-assisted heating.**

A technique for performing microwave assisted organic reactions, termed enhanced microwave synthesis (EMS), has been patented by Hayes and Collins [22] and subsequently reported by Hayes [23]. In this process the outside of the reaction vessel is cooled by compressed air, while the microwave irradiation is heating the reactants (**FIG. 1c**). The synergism of volumetric dielectric heating and external cooling enables the reaction to be maintained at a set low point temperature thereby preventing thermal runaway, which could lead to the decomposition of the target molecules. This can result in a significantly higher yield of the target product molecules as well as a cleaner processing chemistry.

Microwave susceptor: Susceptors designed for microwave ovens have the ability to absorb electromagnetic radiation in the 2.45 GHz range and covert this energy into heat, usually by re-emitting the energy in the form of conduction or infrared radiation (**FIG. 1d**). The integration of a susceptor into the microwave process is often termed as 'Hybrid heating'. An example of a microwave susceptor developed for the food industry, are polymer wrappings coated with a thin film of aluminium. [24]. Another example in this sector are paperboard sleeves or pocket coated with a microwave interactive material [25]. A further example of the use of microwave interactive material, is the use of particles of graphite, magnetite, silicon carbide or copper (II) oxide), to porous solid refractory materials that are and microwave transparent [26]. The combination of microwave irradiation and the susceptors stored energy and re-emit as heat facilitating the crisping and browning of food. Outside the food industry, susceptors aid the processing of organic and inorganic materials that are weak absorbers of microwave energy [27,28]. Bhattacharya and Basak [29] have written extensively on the use of susceptors in non-plasma microwave processing of materials. An advantage of mixing the susceptor with the process material is that it enhances direct heating, but at the expense of chemical contamination. In order to prevent this contamination, susceptors can be separated from the processing material or can be placed around the reaction vessel without blocking the microwaves. Alternatively, the removal of the susceptor may require a post-process decontamination stage. The use of a susceptor in some cases may lead to a thermal runaway, due to the continuous increase of the microwave absorption with temperature rise is to use sufficiently low levels of solid or powdered metals to prevent a thermal runaway reaction. However, the addition of metals does not remove the contamination problem.

Microwave photochemistry: Círka and Relich have written extensively on the combination of ultraviolet and visible emission (UV/Vis) light for photochemistry treatments, combined with microwave treatments. Both batch and flow-through processing have been investigated [30]. The approach to generating UV/V in eth microwave oven, is the uses of Electrodeless Discharge Lamps (EDL) filled at low-pressure 10-3 Torr (0.133 Pa) with a mixture of an electropositive gas (He or Ar) and mercury (Hg) vapour with the EDL envelope made from quartz. Under microwave irradiation conditions, the electropositive gas atoms are ionised to produce an avalanche of electrons that collide with the heaver Hg atoms, exiting them from the ground state to an exited state. These exited atoms then relax by producing photons at wavelengths that range from x-rays to infrared. The EDL quartz envelope restricts this electromagnetic to the wavelength range ʎ=200 nm to 700 nm, which corresponds to the Bond Dissociation Energy (BDE) of functional groups (N, O, H, OH) that are bonded to aromatic and long-chain carbon molecules (polystyrene and polyethylene.,). The steric effects within these aromatic and long-chain molecules however affect the BDE of functional group to the parent molecule resulting in a decrease in the bond cleavage wavelength as compared to diatomic molecules [31].

The construction of different EDL topologies enables discrete external UV illumination, internal UV/Vis illumination and/or surround illumination where the electrical discharge fills the internal space of a double walled reaction vessel (**FIG. 2a-c)**. **FIG. 2d** shows a typical filling apparatus for the EDL. Reference [30] tabulates over 30 organic reactions using these three methods of microwave photochemistry for both batch and flow-through processes.

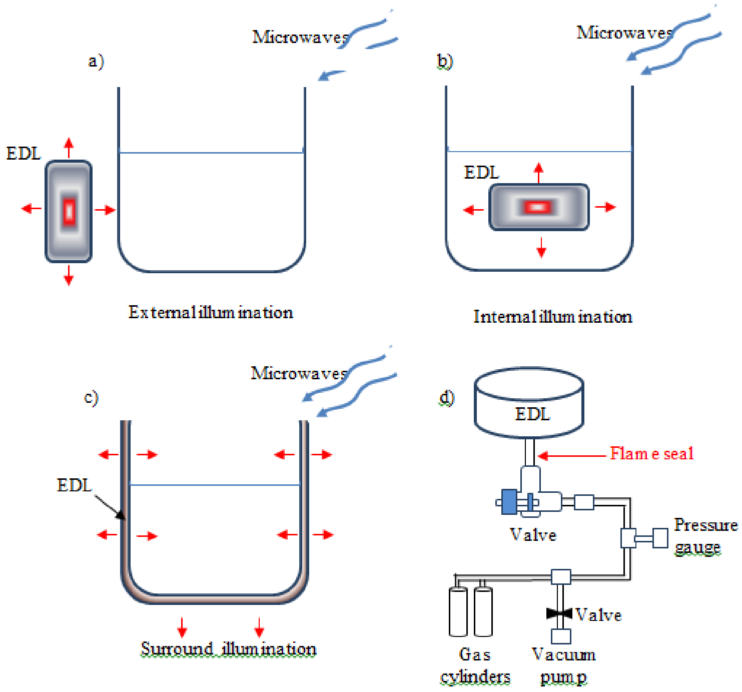

FIG. 2. **Configurations of electrodeless discharge lamps (EDL) illumination of reactor vessel: a) external, b) internal, c) surround, d) Typical EDL filling apparatus.**

Microwave irradiation of water and it use: Industrial microwaves treatments are employed in the decontaminated of polluted water and to treat water for used in the pharmaceutical [32,33] and horticulture sectors [34,35]. Microwave ovens have been used for localized treatments also. For example, steam generated through the microwave treatment of water has been used in the sterilization of dentures [13]. The technology has also been used to sterilize microwave proof plastic containers [15,16].

Within the pharmaceutical industry, Wong et al studied microwave oven (Sanyo EM-GA), for the irradiation of water and its influence on the dissolution of drug tablets [32]. Their results showed that microwave-treated water increased the dissolution propensity of both hydrophilic and hydrophobic free drugs and drugs encapsulated in calcium. The reaction pathways associated with this process was the increase in pH level within the microwave-treated water (due to the removal of dissolved carbon dioxide), which leads to an increase water up-take within the drug tablet.

Karthikeyan et al reported on an integrated microwave-ultraviolet oven for the treatment of contaminated water: the contaminate being aqueous p-cresol (C_7H_8O) [33]. Brief details of the converted microwave oven (Kenstar-Ken chef, model No. MWO 9807), rated at an output power of 300 W is as follows. The mercury lamp with a wavelength of 352 nm at 6 W (Heber Scientific, India) is aligned along the longitudinal axis of a reactor quartz cylinder (50 outer diameter, 18 mm inner diameter and 240 mm in length) that passes through the length of the MRC. To prevent microwave irradiation damage to the lamps metal electrodes the electrodes, they are positioned outside the MRC. The contaminated water flows through the reactor, where it is irradiated with the microwaves and UV emission. Using TiO_2 as a catalyst a 2 to 3-fold. Greater efficiency in the removal of TOC (Total Organic Carbon) was achieved, compared with UV-TiO_2 treatment alone.

Microwave irradiated water experiments of plant seedlings by Gupta et al. was investigated using an IBF microwave oven operating in the continuous mode at power levels of 30 W, 50 W, 70 W and 90 W [34]. Similar treatments carried out by Atallar et al [35] using an unspecified oven operating at 1000 W. In the case of Brassica seedlings [34] the results indicated that plant growth is dependent on both power level and irradiation time. Continuous exposure at power levels above 50 W, with treatment times of more than 60 seconds, was found to inhibit growth. Microwave irradiated power levels of 30 W with irradiation times of 60 seconds, was found in contrast, to enhance germination.

The effect of microwave treated water on the growth of both corn (*Zea Mays*) and pepper (*Capsicum Annuum*) seedlings was studied for 30 days [35]. It was found corn seedlings which had been exposed to microwaved water showed lower growth rates in comparison to the control ones, Corn seedlings when watered with normal water or with water heated on the stove grew faster and have shoot length significantly bigger than the corns which were watered with water heated in a microwave. In contrast, pepper seedlings watered with either microwaved water or non-microwaved water was found to have no significant effects on their growth characteristics. It was concluded that individual plant growth is very specific to microwave treated water.

Ultrasonic assisted microwave oven processing: In addition to UV/Vis, the use of ultrasonic assisted microwave processing is generally associated with the treatment of organic compounds and biomaterials using a fixed geometry waveguide applicator [9]. For the microwave oven this has proved technically difficult and potentially hazardous, as this requires the placement of an ultrasonic metallic horn inside the oven. To overcome this problem, Leonelli and Mason proposed two separate reactors, the first using ultrasound reactor and the second as a microwave oven with a recirculating pump to allow the liquid to be transferred from one reactor to the other [36]. In addition they discussed hybrid systems that have a metallic horn illumining the microwave zone from outside the MRC, and the use of non-metallic horn within the MRC. Moving further away from the microwave oven design, Horikoshi and Serpone [37] reported on the integration of the microwave electrode and ultrasonic homogenizer for the generation of in-liquid plasma.

Microwave oven experiments in school, university and at home

Writing in the journal Bell Jar (in 2001), Hideaki Page was one of the first to discuss the problems encountered when converting a microwave oven into a plasma reactor using an invert glass jar and bowl, as the plasma chamber (**FIG. 3a**) [38]. In this dedicated journal to amateur investigators, the editor responded saying 'that there is a commercial adaptation of a standard household microwave oven that is offered as low cost plasma cleaner and asher.' It took three more years (2004) for university researchers to report on this issue, within an educational environment (Physics Education. IOP publishing Ltd) the principles of microwave oven physics [39], microwave cooking [40] and microwave experiments using metals and light sources [41]. A few years later (2009), Stanley published two review articles aimed at school applications of the technology [42,43]. Throughout [39-43] the emphasis was on how the microwave oven is now part of many students' lifestyles: and how the oven can be a great kitchen-based teaching resource: with the proviso that all safety precautions are followed along with full supervision of the students. The experiments ranged from the: generation of plasma balls, exploding eggs and the creation of soap sculptures, measurement of the speed of light, susceptor in the form of a metalized paperboard box [40], as well as the effect of placing metal objects in the oven and turning it on!

Eduction.com provides a number of microwave school experiments one of which (microwave plant experiment: radish seed germination [44]) encourages students thinking of what happens when microwave irradiated water is used to irrigate seeds and plants. This approach however caused controversy on many internet websites [45].

Harrison described in a Cambridge University project, the conversion of a microwave oven (Samsung MW 76N-B/XEU) for air/argon plasma cleaning of glass and copper surface [46]. In this plasma reactor, a similar plasma chamber design to that of Page (inverted glass bowl/jar with the gas and vacuum ports at its base [38] is used to support a sub atmospheric pressure of 3×10^{-3} to 10 mbar (0.3 to 1000 Pascal). The reported microwave power was 800 W. A similar chamber was also used by Seller in his PhD thesis, where he investigated "The effects of plasma etching substrates on the performance of group 11 metal thin films" [47].

YouTube video-sharing web site is an important recourse for amateur microwave oven experiments, many of which are wacky and dangerous. Reference [8] lists a number of microwave oven postings that range from the generation of plasma balls to converted magnetron experiments. The authors of this article and some of the video authors clearly warn that these experiments should not be repeated at home. One posting from the Centre National de la Recherche (CNRS)-France gives a very informative presentation on the use of a converted microwave oven (again using Page's glass bowl design for plasma cleaning of glass surfaces, but this time the plasma chamber is turned through 90 degree [48] (**FIG. 3b**).

To conclude, plasma chambers constructed from an inverted glass bowl or jar with its gas and vacuum port at its base is a popular design: presumably due to its ease of construction, including no need to rebuild the microwave door. This simple design however has the disadvantage in that it cannot accommodate in-liquid plasma processing without further design change. These design changes for are given in section 4.

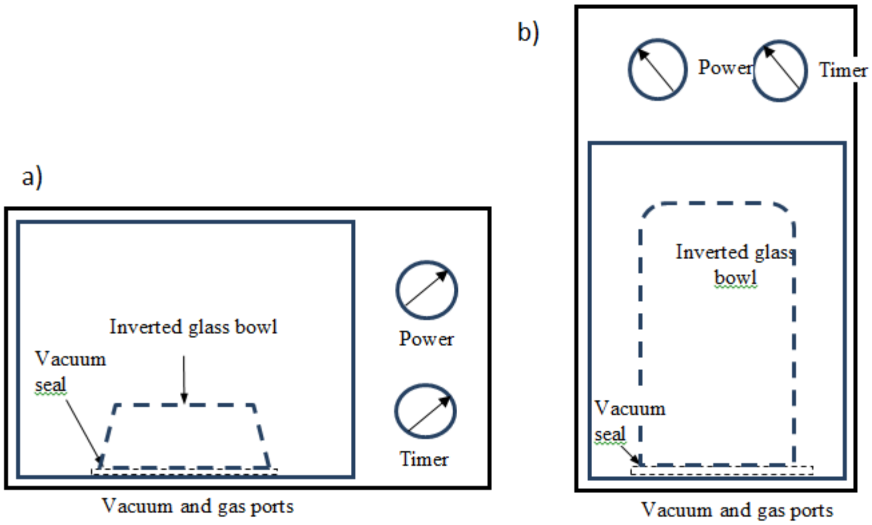

FIG. 3. **a) Front view schematic of [38,46,47] converted microwave oven, b) Front view of CNRS [48] converted microwave oven.**

Microwave oven plasma processing

Microwave oven plasma processing of PDMS: In late 1990s the silicon elastomer PDMS ($(C_2H_6OSiO)n$) was widely selected as the precursor of choice for the fabrication of microfluidic devices: Duffy et al [49], Kim et al [50] and McDonald et al [51]. Arguably, the choice of PDMS, comes from is its conformal sealing properties, high electrical break down voltage, transparent property in the optical wavelength that extends in to the UV wavelength of 240 nm and is chemically inert to most organic solutions. In addition, PDMS is a forgiving material to work with, when compared to silicon and glass being the bulk fabrication material and requiring less capital out. One of the challenges in the fabrication of these devices is to seal the three-sided fluidic channel to its capping layer to a degree that it can withstand the design working pressure without leakage. Usually the functional complexity of the device and the operating pressure dictates whether an irreversible seal that uses a plasma-cleaning step prior to conformal bonding or a reversible is used. The irreversible sealing approach can achieve a working pressure (30 to 60 psi (138 to 413 k Pascal) as compared to reversible sealing (5 psi (34.4 k Pascal)) but at the cost of not being able to rebuild and reuse the device, when it becomes clogged. During this time period (1998-2003) the plasma cleaning step was performed using a radio frequency inductive oxygen (O_2) plasma system (PDC-23G, Harrick, Ossining, NY) [49] or long process times (10 minutes) in a high voltage corona discharge system [50]. Both of which generated short-lived hydrophobic properties.

To circumvent these early oxygen plasma problems Hu et al [52] employed a custom built 200 W Hg-lamp UV-grafting with an exposure time of 1 to 4 hours to apply a hydrophilic coating on to the PDMS surface prior to reversible sealing. Two years later Berdichevsky et al reported on a UV/ozone grafting treatment to impart a hydrophilic coating on the PDMS surface [53]. The addition of ozone (O_3) reduced the processing time from 120 to 30 minutes. To explain the increased efficiency of the process a three-step grafting process was proposed: first the UV/ O_3 apparatus (Jelight, Irvine, CA) generated O_3 through the interaction of UV light (λ=185 nm), with oxygen in the air. Second UV light at λ=254 nm interacts with the O_3 to remove hydrocarbons containing moieties at the PDMS surface. The third step involved UV light at λ=185 and 254 nm converting the PDMS surface from CH_3-Si-O to Si-O-Si networks.

Ginn and Steinbock reported on plasma cleaning of PDMS surfaces using a domestic microwave oven (Amana, ACM2160AB) [6]. In their oven the turntable was replaced with a removable evacuated (~10-3 Torr (0.133 Pascal)) desiccator plasma chamber, which contained the PDMS samples along with an aerial antenna (**FIG. 4a**). With the chamber placed within the oven, the residual air forms the plasma working gas, when the microwave power is applied (1100 W). However due to static air pressure the process time is limited to a few 10s of seconds. Their water contact angle measurements revealed that a dramatic reduction in the water contact angle of PDMS, from 112 ± 2^{o}C to less than 15^{o}C, with plasma exposure times of more than 25 seconds. The polymers hydrophobic recovery was reported to be 2 to 3 hours. Optical emission spectroscopy (350 nm to 900 nm) of a microwave oven air-plasma (Barnes et al [54]) reveals the dominant emission comes from the second positive system of molecular N_2, the first positive nitrogen ion N^{+2} and atomic oxygen. These emissions combined with the absence of NO_2^{*} continuum, indicate a gas rotational temperature of the order of 500 K to 1000 K [55].

The hydrophobic to hydrophilic property change for PDMS has been widely reported to be associated with enhanced levels of oxygen functionality on the polymer surface after plasma treatment [56]. Hui et al developed the microwave oven technique further by using a similar removable evacuated glass jar but replacing the residual air with O_2 at a pressure of 2-5 Torr (0. 26 to 0. 66 k Pascal) [57] (**FIG. 4b**). The O_2 replacement extended the microwave irradiation time 30 to 60 seconds at an estimated (calorimetric method) applied power of 30 W to 90 W. Their PDMS experiments also found that they could produce an irreversible seal between two PDMS slabs. A possible explanation for this strong adhesion is that after microwave plasma treatment, it is now generally considered that when two highly hydrophilic PDMS surfaces are brought together, that their oxygenated repeat units facilitate the formation of interfacial Si-O-Si covalent bonds as of water molecule are displaced from the interface. Hence, this covalent bonding is the main reason for the irreversible conformal sealing. Whereas non-plasma treated surface must rely on weak Van de Waals forces at the conformal interface and therefore produces a reduced bond strength that provides the reversible seal [58,59].

A low cost wet chemical alternative to the plasma treatment of polymers to enhance their surface energy has been reported by Koh et al. They used a Piranha solution which consists of a mixture of concentrated sulphuric acid and hydrogen peroxide [60]. Handling and disposal of the aggressive corrosive solutions and inherent PDMS swelling side effects however, does not provide a safe and satisfactory alternative to the microwave oven approach: particularly when rapid prototyping or proof-of-principal studies are required in the development stage.

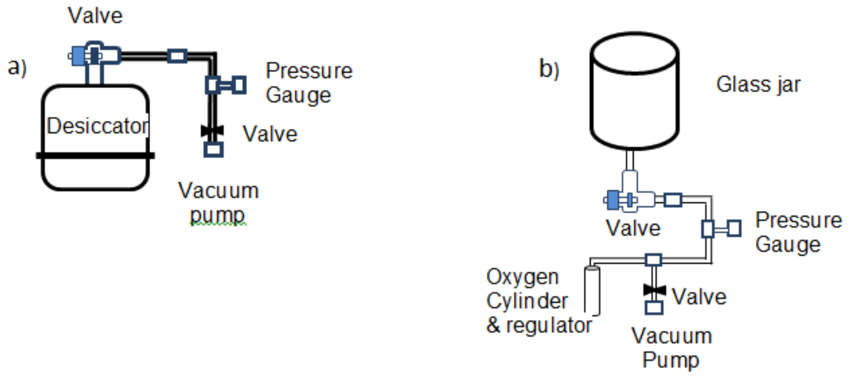

FIG. 4. **a) Desiccator plasma chamber with pumping system [6], b) evacuated glass jar with vacuum pump and O_2 refill systems [57].**

Microwave plasma processing of carbon nanostructures: There have been at many studies reported on the use of microwave oven for rapid prototyping and proof-of-principle production of carbon-based nanomaterials [7,61-68]. The process includes the synthesis of a wide range of carbon allotropes, ranging from: single and multi-walled carbon nanotubes (CNT and MWCNT, respectively), Onion-Like Nanostructure’s (OLNs), fullerene and graphene. To help the reader, **TABLE 1** gives the basic process window parameters for these processes. The microwave oven approach outlined here differs greatly from the high-end single use plasma systems: for example, magnetron sputters systems [61].

In-liquid chemistry: Nomura et al [7] have described the use of a converted microwave oven (output power of 750 W), for in-liquid plasma decomposition of n-dodecane (molecular formula: $C_{12}H_{26}$), to simultaneously produce hydrogen gas and carbide. The reaction is performed within a sealed Pyrex vessel containing 500 ml of n-dodecane liquid, with one or more aerial-antenna igniters. In addition, two silicon/PTFE tubes, inserted through the top of the cavity, for supplying gas (argon) and to collect both the spent argon and by-product gas at a working pressure close to atmospheric pressure. In a follow-up experiment, using the same apparatus and microwave power (750 W), they produced MWCNTs with cyclohexane (C_6H_{12}) as the hydrocarbon source [62]. Toyota et al has also used n-dodecane in a converted microwave oven at 500 W to produce diamond films [63]. **FIG. 5** provides a typical representation of the reactor.

Singh and Jarvis reported the generation of carbon-nanostructures within a continuously pumped 3-port reaction flask (made from borosilicate glass and 1000 ml volume), placed within a 1000 W rated microwave oven [64]. To support the vessel and facilitate access to it the oven, the oven door is replaced with an aluminium plate (of the same size as the door), that has three apertures; one for each flask port. With the flask supported, it is evacuated from the outside of the oven using one port, while the other two ports provide carrier gas (hydrogen) and the selected hydrocarbon precursor based on their hydrogen-to-carbon ratio (ethanol (C_2H_6O), xylene (C_8H_{10}) or toluene (C_7H_8)). To enhance the reaction a 2 mm diameter aerial-antenna igniters mounted on a stainless-steel base within the reaction flask is used. As no vacuum pressure or microwave power was reported it is assumed that the flask was sub atmospheric and the microwave power was at maximum (1000 W). Their microwave process revealed that selectively between OLNs and CNT is achievable. For the production of CNT an ethanol (C_2H_5OH) solution with the heterocyclic compound thiophene (C_4H_4S) as an additive plus a number of aerial-antenna igniters. For growth of onion-like nanostructures, either toluene ($C_6H_5CH_3$) or xylene ($C_6H_4(CH_3)_2$) is used without an aerial-antenna igniter.

TABLE 1. **Basic process window parameters for microwave oven plasma synthesis of carbon nanostructures.**

Microwave oven	Hydrocarbon source	Susceptor/ igniter	Product	Stated MW power (W)	Process time	Ref
In-liquid chemistry						
Not given	n-dodecane	igniter	carbides	750	28s	7
Not given	cyclohexane	igniter	MWCNT	750	28s	62
	n-dodecane	igniter	diamond film	500	10 minutes	63
Not given	ethanol xylene toluene	igniter	CNT, OLNs	1000	Not given	64
Solid-state chemistry						
NEC N920E	polystyrene	aluminium or graphite	Fe_3O_4/carbon composite	700	90s	65
NEC N920E	polystyrene polyethylene	aluminium	graphene sheets	700	120s	66
DEC18E2	naphthalene	graphite	OLNs	1800	15, 30, 45 and 60s	67
Not given	polyethylene	aluminium	CNT, MWCNT	Not given	Not given	68
Samsung M539 MAN200405W	rice husk	aluminium	CNT	600	38 minutes	69

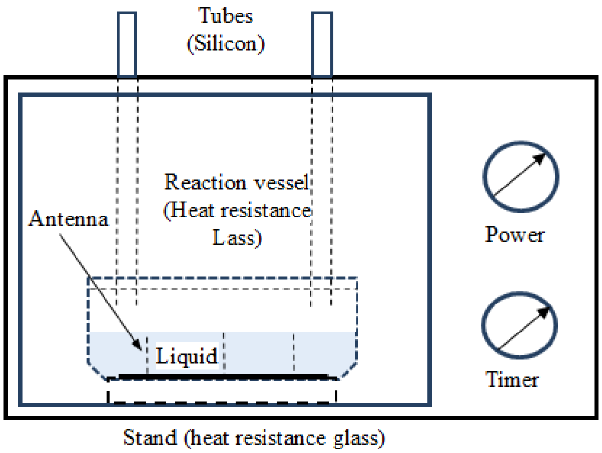

FIG. 5. **Front view schematic of the converted microwave ovens for in liquid plasma processing [7,62,63]. For clarity, the auxiliary gas lines outside the ovens are not shown.**

Solid-state chemistry: Hojati-Talemi et al reported on the use of a domestic microwave oven (NEC N920E) for the preparation of Fe_3O_4/carbon composite nanoparticles [65]. Their process used a polystyrene ($(C_8H_8)n$) mixed (10% by weight) with magnetite nanoparticles (diameter 20 nm-50 nm) that also work as a subsector: all of which was prepared in a solution of tetrahydrofuran ($(CH_2)_4O$) then dried to a composite. The mixture was then placed inside an evacuated (1 mmHg (133.3 Pascal) quartz tube that contained a plasma igniter (block of aluminum or graphite). With the tube placed in the oven and microwave irradiated at 700 W for 90 seconds. Once the plasma is generated the emission changes from a bright emission to a yellow color as the polymer starts to degrade. Post plasma treatment analysis (Raman spectra analysis, TEM imaging and magnetization studies) revealed a formation of a graphite coating around the magnetite without any observable lost in the magnetic properties of magnetite In their conclusion they postulate that the graphitization of the polystyrene may be used as a protective barrier around the magnetite thus making the particles electrically conductive which may allow subsequent modification of the nanoparticles.

Hojati-Talemi and Simon, used their same apparatus to demonstrate, that by increasing the hydrogen: carbon ratio in the hydrocarbon source from 1 for polystyrene to 2 for polyethylene ($(C_2H_4)n$) the product changed from OLNs to graphene sheets [66]. Equation 1 and 2 shows a general reaction of these processes.

$$(\mathrm{C}_8H_8)n \xrightarrow[plasma]{} OLNs \qquad (1)$$

$$(\mathrm{C}_2H_4)n \xrightarrow[plasma]{} graphene\ sheet \qquad (2)$$

Bajpai et al reported the on the production of OLNs spheres a microwave oven ($DEC_{18}E_2$, ACP, rated at 1800 W) [67]. Their process used graphite as a microwave absorber and naphthalene ($C_{10}H_8$) as the carbon source. Using a 200 mg mixture at a ratio of 1:2 within a quartz vial placed in the oven produced violent arcing reaction when irradiated at full power for a process time of 1 minute. Equation 3 provides a representative reaction for this process. Outside the quartz vial they were able to deposit onion-like rings onto ceramic substrates. Here again the microwave induced arcing is intense and vigorous.

$$(C_{10}H_8)n \underset{arc}{\longrightarrow} OLNs \tag{3}$$

Kure et al also reported on the uses of polyethylene as a solid hydrocarbon source to prepare CNTs within a microwave oven [68]. Although they did not name the microwave oven used, their starting material consisted of 100 mg of polyethylene placed on aluminium foil, along with a silicon substrate coated with iron (III) nitrate. These where placed within an 1860 ml quartz tube reaction chamber which was evacuated to a pressure of 0.81 mbar (81 Pascal). The prepared reaction chamber was then placed in the microwave oven and irradiated at 600 W. As reported by other authors [65,66], once the plasma is turned on a purple emission is observed but changes as the decomposition process begins. The reactants where allowed to reach a synthesis temperature of 760°C, at which point the microwave power was turn-off and then allowed to cool to room temperature: from turn-on to collection of the cooled products took approximately 1 hour. Their analysis revealed their products were a mixture of CNTs and MWCNTs with diameters in the range of 1 nm to 25 nm and lengths of about 0.85 μm.

Rice Husks [RH] are a major by-product of the rice milling industrial and attempts to convert this waste into activated carbon using microwave irradiation has been explored [69]. Anaswi et al reported on plasma induced synthesis of carbon nanostructures from waste RH, which contains both cellulose and lignin, using a Samsung microwave oven (M539 MAN200405W) operating at 600 W for 38 minutes [70]. Their experiments revealed that the vacuum pressure 1 mbar (100 Pascal), played a critical role in the decomposition process. The incorporation of the organometallic compound ferrocene ($(C_5H_5)_2Fe$) was also found to have a catalytic role in the plasma induced reaction. To account for the relative long process time, the authors postulate the tip down growth mechanism where hydrocarbon decomposition begins on the outside of RH and then diffuses through pores (generated by the microwave irradiation process [69]) and to the bottom of the husk. They also concluded that the use of waste RH to achieve high-value carbon nanostructures reduces the environmental impact caused by the world’s huge production of RH.

Discussion

This work has reviewed the domestic microwave oven as a rapid plasma-processing multi-task prototyping tool. This microwave oven approach enables a wide-ranging study to be completed that informs both pilot and full-scale synthesis. The ovens low cost of ownership, small footprint coupled with limited hazardous wet chemistry use marks it as an ideal candidate for selection in the new manufacturing concept of ‘process intensification’ [17]. The replacement of RF discharge and corona discharge in the manufacture of PDMS-base microfluidic devices provides a good example of ‘process intensification’. The review includes synthesis and enhanced microwave synthesis of organic compounds, microwave susceptor, microwave-UV treatment and experiments at school and at home. The name, where possible, of the microwave ovens are reported, along with their process widow parameters. In all cases, the power source is the package cavity-magnetron.

To exemplify the multi-task capability of the microwave oven, two contrasting plasma domain processes are studied. These are the sealing of PDMS used in the production of microfluidic devices and the processing of carbon-based nanomaterial's.

The first domain examines, microwave, UV and UV/O_3 irradiation for reversible and irreversible sealing of PDMS at the centimeter scale. To control the PDMS surface temperature within the oven and therefore the ability to delineate between reversible and irreversible sealing, the technique of forced air-cooling of the reactor vessel [22,23] along with selected UV wavelength irradiation [30,33], are candidates for future study.

In the second domain, eight examples of carbon-based nanostructures processes are discussed, where the carbon allotrope products ranged from grapheme sheet, CNT, MWCNT and OLNs plus the graphite encapsulation of magnetite. Within the solid-state chemistry section (4.2.2), product selectivity between CNT and OLNs using a different ratio of hydrogen to carbon in the hydrocarbon precursor is established. Moreover, grapheme encapsulation of magnetite and other compounds, including medical drugs, where the external encapsulation layer provides protection to the internal compound may be achievable. Fullerene cages (C60-70 having a typical diameter of 0.7 nm) are too small to encapsulate magnetite (and medical drugs) therefore CNT and OLNs are required. Equation 4 shows one such feasible stoichiometric plasma induced reaction between polystyrene and magnetite. In this equation, one CNT encapsulates one magnetite molecule with the excess hydrogen liberated as hydrogen gas.

$$(\mathrm{C}_8 H_8)n + Fe_3O_4 \xrightarrow[plasma]{} CNTFe_3O_4 + 4nH_2 \qquad (4)$$

To obtain useful yields of the targeted product that does not contain contamination from metallic substrates requires an in-liquid environment where passive aerial-antenna igniters are used. The apparatus in reference [7,62,63] may be used where the magnetite nanoparticles are mixed with the hydrocarbon precursor within a solvent. Moreover, the technique of forced air-cooling of the reaction vessel [22,23] to control the magnetite surface temperature, and Hg-EDL [31] and Hg lamps [33] to enhance hydrocarbon bond cleavages are easily utilised.

One of the futures challenges that face the microwave oven as a rapid processing tool for production of carbon allotropes at the nano-scale, is the successful integration of an ultrasound applicator. If this is achieved, dual microwave heating and sonochemisty (through harnessing the energy within the cavitations bubbles), provides a processing environment that controls the type and size and carbon allotrope. From an electrical engineering point of view a diplexer [71], may be employed to achieve this end. In this circuit; the low-pass filter in the range of 1000 kHz is used to protect the ultrasound transducer [37]. The TE10 waveguide is used protect the magnetron from the ultrasound energy and; the MRC is where the ultrasound assisted process takes place. An example of such a diplexer circuit within a microwave oven is schematically shown in **FIG. 6.** Here it is shown that the ultrasound traduce and low-pass filter is placed below the in-liquid reaction vessel with a microwave screen manufactured in the MRC wall to prevent microwave irradiation of the low-pass filter (the screen being typically an array of 2 diameter holes arranged at 6 mm centers). The ultrasound coupling to the reaction vessel is determined by the probe geometry and coupling efficiency through the microwave screen. Another coupling mechanism could be for the ultrasound probe to pass through the MRC wall and encapsulated in the polymer stand, on which the reaction vessel is placed.

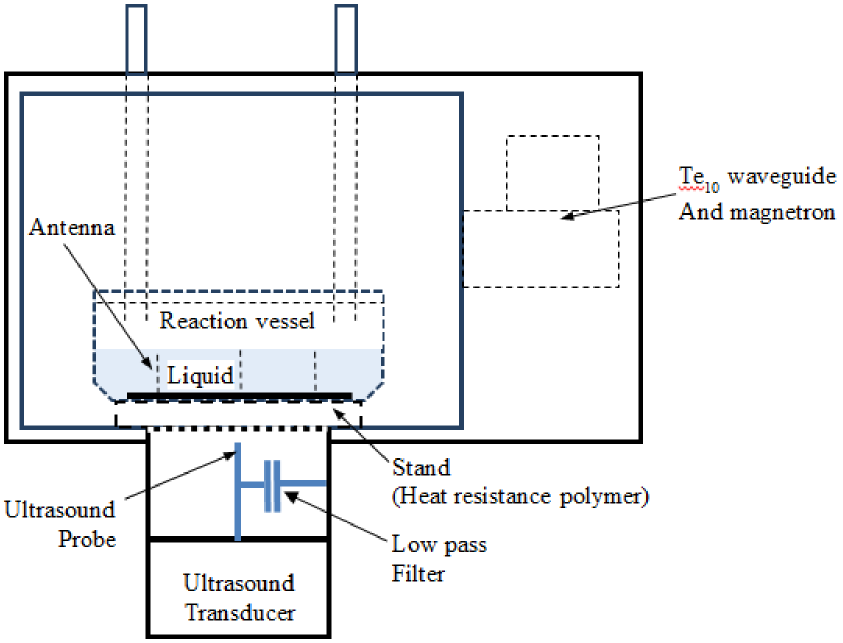

FIG. 6. **Front view schematic of the converted microwave ovens for in-liquid plasma processing with integrated ultrasound transducer with low-pass filter. For clarity, the auxiliary gas lines outside the ovens are not shown**.

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