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Impact of non-thermal plasma on the abatement of Perfluorinated compounds present in minute quantities in water effluents

Muhammad Jehanzaib Khan[1], Vojislav Jovicic[1,2*], Ana Zbogar-Rasic[1], Nataliia Fedorova[1, 2], Alexander Poser[3], Peter Swoboda[3], Antonio Delgado[1, 2]

[1] *Institute of Fluid Mechanics (LSTM), Friedrich-Alexander University (FAU), Erlangen, Germany*
[2] *Erlangen Graduate School in Advanced Optical Technologies (SAOT), Germany*
[3] *R&H Umwelt GmbH, Schnorrstraße 5a, D-90471 Nürnberg, Germany*

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Abstract

Based on the strongest C-F bonds backbone, Perfluorinated compounds (PFCs) are manmade chemicals that are omnipresent due to their immense use for the production of various products. Currently, these are also addressed as global contaminants, and are present on the word market since middle of the 20th century. Non biodegradability and high half-life times generate serious adverse effects on humans and wild life. This calls for remediation. Updated lifetime health advisory, and water quality standards have been issued for PFCs by different US and European environmental agencies, but the conventional techniques for remediation are either inefficient, or turn out to be uneconomic, especially dealing with extremely minute quantities present in effluents.

The present study copes with the reduction/removal of low concentrated (<1 µg/l) PFCs from waste water, using non-thermal atmospheric plasma (NTAP). The research experimentally investigates influences of different parameters e.g. plasma gases (air, O_2 and N_2), treatment times (up to 10 min.) and nozzle/reactor sizes on the reduction of PFC concentrations.

Promising results were obtained with NTAP exposure on the PFC concentration levels. In less than 200 seconds, the concentration thresholds of below 50% of initial PFCs concentration was reached in most of the tests within the shown experimental setup. The final concentration of PFCs was strongly dependent on the tested parameters. The decreasing pH value of the water was found to be a limiting factor for the lower PFCs concentration threshold.

Introduction

Perfluorinated compounds (PFC's) are the compounds that are known due to their extreme stability and their useful unique physical and chemical properties in the production of a variety of products [1]. These human made organofluorine chemicals are omnipresent and are of concern due to their non-biodegradable nature and bioaccumulation [2]–[5]. PFC's have been manufactured for over 50 years [6]. Their chemical structure is based on long C-C chain, with a general formula $F(CF_z)_n$-R, having several C-F covalent bonds that constitute one of the strongest and stable covalent bonds in chemistry (dissociation energy ΔH = 450 kJ/mol) [7]. The wide spread use has resulted in a release of PFCs into the environment either from direct sources such as the manufacturing of PFCs and the use of various products using PFCs or from indirect sources like reaction impurities or precursor degradation [4]. The global ecosystem that is responsible for supplying millions of humans

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worldwide with drinking water has been severely affected due to PFCs' fast water dissolution properties [8]–[10]. Details on PFCs' nature, toxicity, occurrence and potential effects on human health can be found elsewhere [11], [12].

Their chemical stability, non-wettability, high fire and weather resistance, hydrophobic and oleophobic nature prompted their abundant use in various products [9], [13]. The most common applications of PFCs are the production of firefighting foams, detergents, carpets, floor polishes, insecticides and semiconductor industry etc. [14]–[16]. PFCs including CF_4, C_2F_6, C_3F_8, CHF_3, etc. are extensively used as etching and cleaning gases in semiconductor industry especially and contribute to earths greenhouse effect with relatively high global warming potential [17], [18]. At present, PFCs occur in diverse environmental compartments at concentrations ranging from pg/kg levels to µg/kg levels. In the context of risk analysis, Perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS) have attracted most attention, because these compounds contribute highly to the environmental contamination by PFCs [4], [9].

PFCs show high resistance against their degradation using advanced oxidation processes, acids, bases, photolytic processes and hot water treatments [19], [20]. Strong C-F covalent bonds make them resistant against microorganisms and they are persistent and stay in environment longer [7]. Longer half-life times in humans (2-8 years) and surface waters (41 - 92 years) have attracted strong scientific and regulatory attention worldwide [21], [22].

Our eco system has increased levels of PFC contaminates due to its high dissolvable proper- ties. The waste water from different industries is the biggest source of contamination with PFCs [15], [23]. This causes concentrations of PFC from ng/l to µg/l in the ground water [24].

Mhadhbi et al. and others reported the presence of PFCs inside wildlife species, including marine mammals, fish, birds and shellfish etc [25], [26]. Their presence in fruits and vegetables along with their bio accumulative nature can potentially be harmful for humans, resulting in cancer risk, thyroid diseases and weakening of the immune system [27], [28]. Different legislations and initiatives has been taken to reduce the concentration of PFCs in the environment. Stockholm convention and the restriction imposed for the phase out of PFC production by major producers in US by EPA are the key step towards its concentration depletion [29].

Different countries and their respective environment protection authorities have taken notice and defined the permissible limits of PFCs (PFOA and PFOS) in µg/l or µg /kg. According to European environmental quality standard (EQS), the permissible standard is 0.00065 µg/l and this has to be met by 2027 [30]. In the case of the USA, in 2016 the EPA issued Lifetime Health Advisories for exposure from drinking water of 0.070 µg/l for PFOA and PFOS (individually or combined). Some US states developed their own guidelines for PFC presence in ground waters; e.g., Maine set concentration guidelines to 0.060 µg/l for PFOA and 0.100 µg/l for PFOS [31].

Advanced oxidation technology utilizing the OH radicals can't play a role in PFC degradation due to higher dissociation energy for C-F covalent bond [19]. On the other hand, various methods such as UV induced photo chemical reactions [19], [32], VUV irradiation [33], [34], Sonochemical reaction [35]–[37] and sulfate radicals degradation [19], [38] not only have limitations in degradation but also turn out to be economically unfeasible. Ion exchange resins and the active carbons use the principle of binding PFC through exchange of ions and adsorption respectively and to the date remain as the largest industrial remediation for PFCs [39]–[42]. For a complete destruction, the captured PFCs must be incinerated at higher temperatures above 1000 K [43] thus turning these processes uneconomical.

Non thermal plasma has been studied by different authors as an alternative for the degradation of PFC [44]–[47]. The drawback of all the previous studies on PFC removal lies in the fact that they focus on concentration levels above 1 µg/l. On the other hand, most of

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the demand is to find a remedy for minute levels of PFCs present in water. At these minute levels of contaminants the conventional processes become uneconomical or out of range, urging an investigation of alternative methods.
Plasma is the ionized gas, a collection of ions, electrons and high energy species, capable of initiating chemical reactions. This was first discovered by a physicist Irving Langmuir, in year 1928 [48], [49]. On the basis of the ionization degree of the gas ranging from 100 % (fully ionized) to very low values (partially ionized), the term plasma is classified as thermal and non-thermal plasma. In thermal plasma, all the species present in the gas, e.g. the electrons and ions, have the same temperature ranging from 4000 K to 20000 K [44], [50], [51]. On the other hand, the non-thermal (cold plasma) is produced using less power and the electron temperature is much higher than that of the bulk molecules present inside the gas [44], [49]. The presence of these high energy species, capable of generating the chemical reaction is one way to introduce the advanced oxidation process (AOP), inside the process. Especially, the cold plasma is of much interest in food industry due to its lower temperature.
The goal of the present work was to investigate the potential of the NTAP for degrading very low concentrations (below 1 µg/l) of PFCs (with the emphasis on PFOA and PFOS) from contaminated water samples (batch treatment) originating from the soil cleaning processes.

Experimental design

The water samples contaminated with PFCs were investigated by treatment with non-thermal atmospheric plasma (NTAP), using various plasma gases (air, oxygen, and nitrogen), treatment times (1-10 minutes), plasma nozzles and reactor sizes.
Figure 1 shows the process flow diagram for the treatment of PFC contaminated water with NTAP. This consists of a plasma device (plasma beam [52]), a mass flow controller, a reactor with plasma nozzle for water treatment and an in house designed water cooling system, which keeps the reactor at constant temperature (20-30 °C).

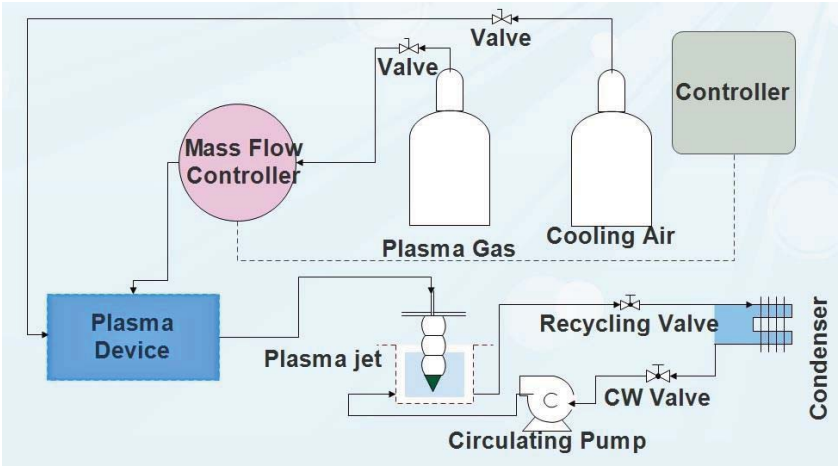

Figure 1 Process flow diagram for non-thermal atmospheric plasma treatment.

The plasma generator consists of two units. In the first unit, high voltage is generated and then the gas for the production of plasma is passed between the two high voltage electrodes in the second unit of the plasma device. The plasma generator used for the current study, has a power of 300 W and a frequency of 20 kHz. Plasma is formed when the activated particles are excited by high voltage electrodes and are pushed away in the form of a jet through the plasma nozzle.
The types of plasma are strongly dependent on the temperature and species characteristics. The plasma in the range of 200-500 °C is called as NTP and contains only the high energy species in comparison to the active plasma where the temperature of the bulk molecules

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are much higher. By interaction of the NTP between the plasma species at the nozzle outlet and the atmospheric air, a strong oxidizing medium is formed that is responsible for initiating further chemical processes within the system.

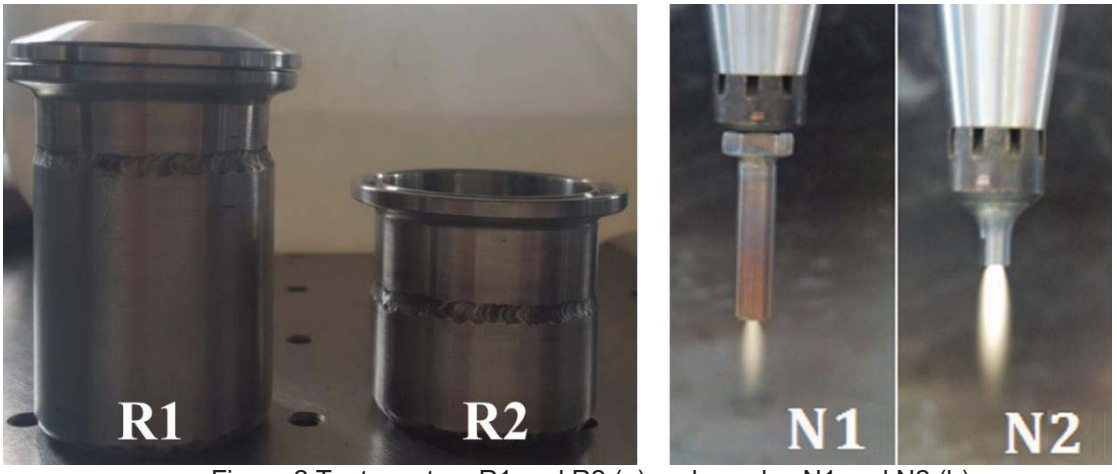

Figure 2 Test reactors R1 and R2 (a) and nozzles N1 and N2 (b).

The first part of the present study investigates the influence of NTAP on the degradation process of PFCs in different reactors (R1 and R2). The outer diameter is d_o = 4 cm, the wall thickness 3 mm, and the heights are L_1 = 56 mm and L_2 = 36 mm, respectively. Initial water sample volumes were V_1 = 45 ml within the reactor R1 and V_2 = 35 ml within the reactor R2. Both reactors were kept at constant room temperature (recorded by K-type thermocouple) by a circulating water bath to avoid the influence of temperature on the degradation process. The second part of the investigation was to observe the influence of a plasma jet on the water samples. Two nozzles (N1 and N2), with inner diameter d_m = 2 mm and lengths of L_{n1} = 34 mm and L_{n2} = 24 mm, respectively were used. Due to different lengths of the nozzles the N1 is always placed deeper inside the sample, compared to nozzle N2. The reactors R1, R2 and the Nozzles N1, N2 can be seen in the Figure 2 left and right respectively.

The third part of the study investigates the influence of different gases (air, oxygen and nitrogen) on the degradation of plasma. Here it is important to mention that the mass flowrate for air and nitrogen was set to $\dot{m}_{pl}$ = 15 l/min, which is the minimum flow rate for the plasma device to generate plasma out of it. In case of oxygen, the mass flow rate was set to 20 l/min due to stability problems.

The waste water samples under investigation during this study were provided and after treatment with NTAP were analyzed by an industrial partner (R&H Umwelt [53]). The initial PFC concentration with in the samples was 0.932 µg/l and sum concentration of PFOA and PFOS around 0.579 µg/l.

Performance of experiments

As a first step, repeatability of the measurement results was performed in two separate sets of experiments, with five tested samples in each set, keeping the test conditions constant. In the first set of experiments, water samples were analyzed within reactor R1 and nozzle N1 for 10 minutes, while the second set of experiments was conducted in reactor R2 and nozzle N2 for 5 minutes. Standard deviation for the performed repeatability was observed to be less than 5 %. Besides the evaluation of the repeatability, these experiments were set as a base for further analysis and comparison of tested water with other operating parameters. The other parameters include the treatments time and combination of reactors and nozzles. The treatment times of 1,3, 5, 7 and 10 min was used for reactor R1 and 1, 3 and 5 min was used for reactor R2. Finally, the treated samples were analyzed by industrial partner R&H umwelt [53] by liquid chromatography – mass spectrometry (LC-MS), following

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the German Norm DIN 38407 F42. The analysis bears the uncertainty level of ±5.505% for the PFOA and ±7.740% for PFOS.

Results and Discussion

Figure 3 depicts the repeatability of the conducted experiments. It is based on the two sets of experiments (R1-N1 and R2-N2). During the experiments the concentration of PFOA, PFOS and total PFC concentration was observed and the gas flow rate and treatment times were kept constant during execution of experiments.

The first two columns show the experiments with R1-N1 and last two are with R2-N2. The PFOA and PFOS concentration before and after treatment is shown in column 1 and 3 while the total concentration of PFC before and after treatment within two sets of experiments is shown by 2nd and 4th column. The total height of the columns represents the concentration of PFOA/PFOS and PFC before treatment and the solid column height represents their concentration after plasma treatment. The vertical lines with in the columns show the good repeatability of the experiments.

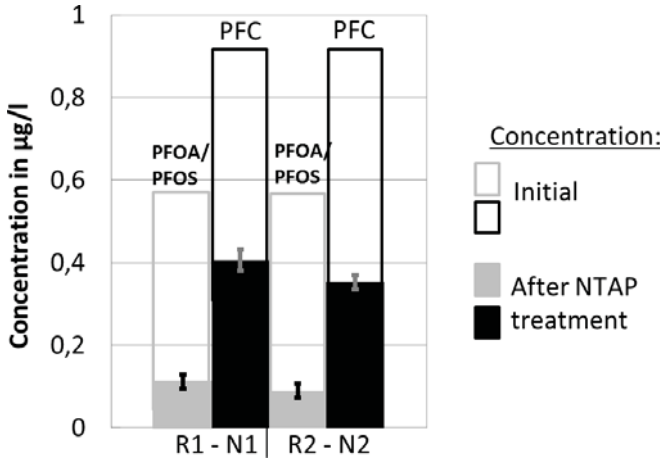

Figure 3 Results of the two sets of experiments (R1-N1, R2-N2), showing repeatability of experiments

The NTAP treatment resulted in a very sharp decline in the concentration of PFCs within the treated water samples. The average PFC and PFOA-and-PFOS end-concentrations after the NTAP treatment were 0.406 µg/l and 0.112 µg/l, respectively (R1-N1), and 0.353 µg/l and 0.090 µg/l, respectively (R2-N2). The reduction efficiency was higher in the case of R2-N2; i.e., 62.1% (PFC) and 84.5% (PFOA-and-PFOS), compared to R1-N1; i.e., 56.4% (PFC) and 80.7% (PFOA-and-PFOS). The reduction is more pronounced for the R2-N2 combination, possibly because the plasma jet, produced by the small nozzle N2, had a good contact with the water sample within the small reaction zone of R2.

Finally, it is noticeable that the concentration reduction was more pronounced for PFOA & PFOS compared to PFC, which further indicates that among a range of different PFCs, a component group exists that is less affected by NTAP compared to PFOA and PFOS.

Influence of operating parameters on the concentration of PFCs

The influence of the reactor size and the treatment times for the fixed nozzle (N1 and N2) was investigated. Figure 4a and 4b demonstrate the results of this study. Results indicate that, independent of the reactor size, the maximum reduction of PFC and PFOA-PFOS occurs during the first minutes of NTAP treatment. Independent of the reaction zone, the treatment of water samples using N1 for longer than 3 min (5 min for N2) doesn't further decrease the PFC and PFOA-PFOS concentrations.

Experimental results (Figures 4a and 4b) also indicate that the contact quality between the plasma jet and the sample volume (i.e., size of the reaction zone) is an important parameter

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for PFC degradation by NTAP treatment. The reduction efficiency is very similar for reactor size-nozzle combinations R2-N1 (highest efficiency), R2-N2 and R1-N1, while the combination R1-N2 is the least efficient. The sample treatment in the larger reactor (R1) using the shorter nozzle (N2) was the least efficient, probably due to a lowest concentration of radicals in the reaction zone and poor contact between the plasma jet and the sample.

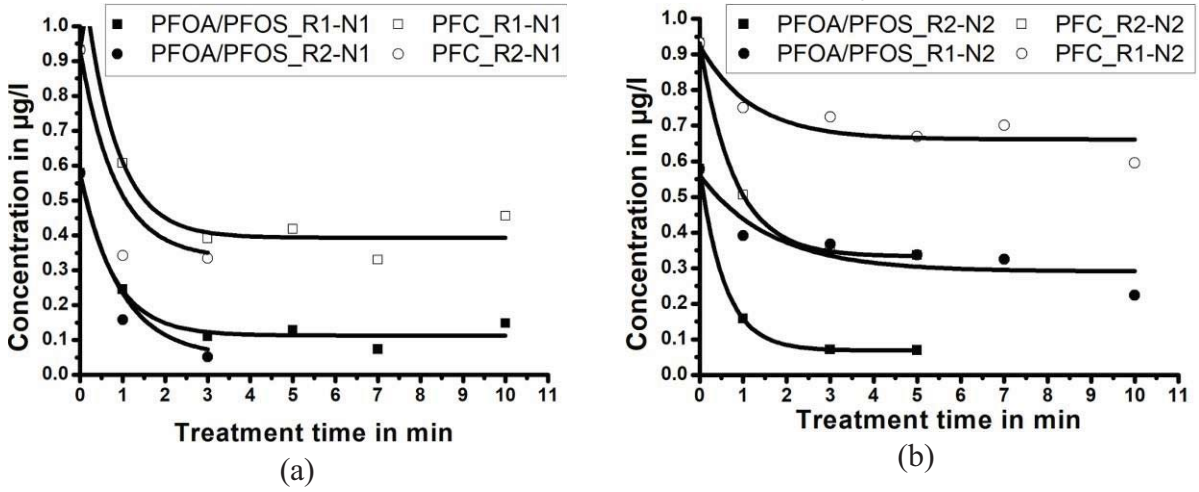

Figure 4 The effect of reactor size and the treatment time on PFC degradation for (a) longer nozzle N1 and (b) shorter nozzle N2

Influence of pH and other plasma gases

As the results in Figure 4a and 4b show that the maximum level reduction occurs during the first few minutes, a pH analysis was performed that shows a concurrent reduction of pH of the samples from pH = 6.8 to the end value of 2.5. Thus, it was assumed that the acidity of the samples negatively influenced the further reduction of the samples.

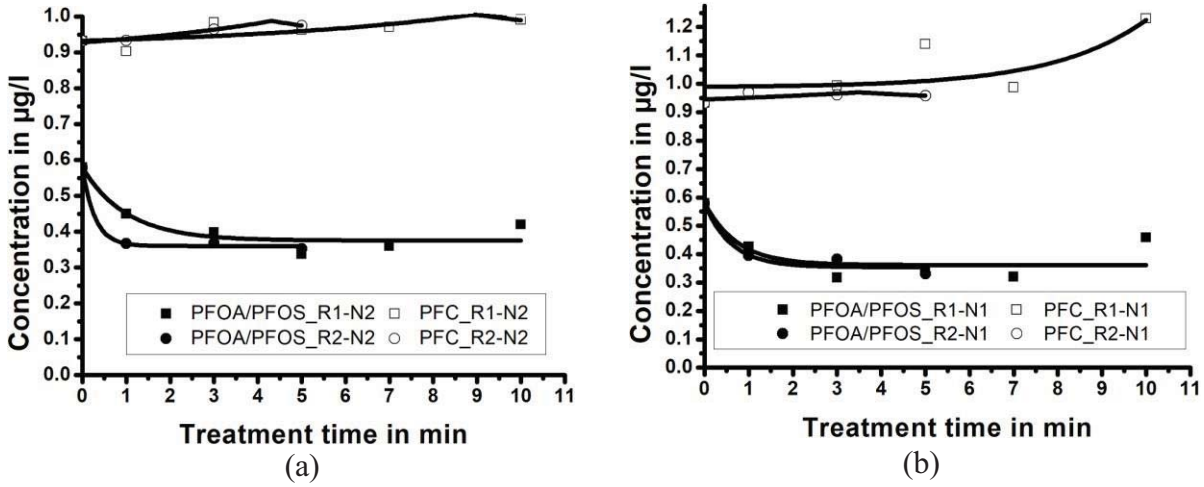

Figure 5 Results of NTAP using oxygen as plasma gas. (a) experiments with N2 and (b) with N1

In order to investigate the influence of the used plasma gas on the PFC reduction in water samples by NTAP, three different plasma gases air, oxygen, and nitrogen were tested. For oxygen the flow rate was fixed at 20 l/min, since at lower flow rates the plasma generator was unstable. Figure 5 shows the PFC and PFOA-and-PFOS concentrations during the sample exposure to the oxygen plasma gas. It can be observed that the concentration of PFOA and PFOS generally decreases from its initial value of c = 0.579 µg/l to the end concentration of c = 0.348 µg/l (treatment time 7 min in R1-N1); i.e., c = 0.353 µg/l (treatment time 5 min in R2-N2). On the other hand, the concentration of PFC increases with the treatment time. Dominant radicals produced by N_2 plasma are excited N_2 molecules in the metastable state. After being injected into the water sample, these radicals disappear

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instantaneously, producing hydroxyl (OH^-) based molecules; e.g., hydrogen peroxide and hydrogen dioxide.

Conclusions

Present paper highlights the potential of NTAP for the treatment of PFC in waste water. Experimental investigations demonstrated that 3-5 min treatment cause a significant reduction of PFC and PFOA-PFOS concentration up to ca. 64% and 90 % respectively. The best results and strongest reduction levels of PFC concentration were observed when the small reactor (R2) was paired with the big nozzle (N1), - the conditions under which a high concentration of plasma radicals can be reached, having a positive effect on C-F bond degradation.

It was also observed that the PFC degradation is limited by the reduction of pH value that can reach as low as 2.3 after treatment but the dilution with fresh sample can overcome this issue and further decrease by NTAP is possible.

Among the tested plasma gases, the best results were achieved with air as the plasma gas. Due to some side reactions, the oxygen increases the PFC concentration, while the effect of nitrogen is less pronounced compared to air. Nevertheless, its implementation on the industrial scale still needs detailed research on different plasma gases and operating parameters.

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