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Role of nitrogen surface legacy and He ion enhanced N-H recombination in the ammonia formation on tungsten walls. A DC glow discharge study.

A. de Castro [a,*] and F.L. Tabarés [a]

[a]*Fusion National Laboratory-CIEMAT, Av. Complutense 40, 28040 Madrid, Spain*

Abstract

Dedicated studies using Glow Discharge plasmas on tungsten walls have been developed trying to understand the surface chemistry and the underlying processes behind the mechanism of the ammonia formation that takes place during N_2 seeded discharges on the inner walls of magnetic nuclear fusion devices. The experiments with pure D_2 plasma irradiation on a cold rolled tungsten wall (previously saturated with a pure N_2 plasma) at 150ºC have shown the formation of deuterated ammonia (mainly as NH_2D and NHD_2) as a result of the interaction between the implanted nitrogen atoms and the impinging deuterium on the surface. Conversely, the symmetric experiment has not shown any significant ammonia production, confirming that the presence of atomic nitrogen (legacy) on the surface is the first and crucial step in the ammonia formation mechanism. The study was completed with experiments that employed N_2-H_2-(He) mixed plasmas trying to understand the role of the helium bombardment (unavoidable in a D-T fusion reactor) in the N-H recombination. The enhanced ammonia generation (up to a 45% factor) observed with increasing helium plasma contents seems to be related with possible changes in the surface roughness and/or structural modification on the tungsten substrate that determines an enhanced recombination.

Corresponding author address:* **Av. Complutense 40, 28040 Madrid, Spain

Corresponding author E-mail:* **alfonsodcc11@gmail.com

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1. Introduction

In the routine operation of diverted magnetic fusion devices, the injection of nitrogen as seeded plasma impurity has become essential to achieve good plasma performance by inducing divertor plasma detachment [1]. The nitrogen molecule acts as a radiative cooler in the plasma edge, with the benefits of improving the confinement due to the associated changes in the edge plasma (pedestal) profile and reducing the heat load to the plasma facing components to an acceptable level compatible with a suitable lifetime for such elements [2]. However, its utilization leads to a significant ammonia formation that has been reported in relevant tokamaks [3, 4]. The deleterious effects of ammonia corrosion on the gas exhaust system, cryo-pumping panels and pumping lines would limit the ITER operational cycle due to the more frequent necessary regeneration and cleaning procedures related with the tritium inventory radioactivity safety issues. The formation of ammonia takes place on the surface of the metallic wall materials that surround the plasma in a heterogeneous plasma-activated catalytic process.

In this work, direct current Glow Discharge plasmas (dc-GD) have been used to simulate the plasma-surface processes in diverted N_2 seeded plasmas that determine the ammonia formation on tungsten walls. In particular, the basic surface chemistry (N-H recombination processes) was studied, emphasizing in the importance of the nitrogen presence (legacy) on the surface to produce ammonia. The role of nitrogen/hydrogen depth implantation on the two different possible recombination reactions (Langmuir-Hinselwood [L-H] and Eley-Rideal [E-R]) was investigated by means of alternate (D_2 or N_2) dc-GD plasmas performed on a tungsten wall previously saturated with N_2 or D_2 dc-GD plasmas, respectively, and their ability to produce ammonia. Furthermore, in an hypothetic reactor environment, the presence of helium as intrinsic impurity, that will be generated in the D-T nuclear reactions, will be unavoidable and must be taken into account. Consequently, the experimentation was extended to simultaneous dc-GD N_2-H_2 plasmas where also helium was introduced. The ammonia formation yields have been deduced from differentially pumped mass spectrometry measurements carried out on a residual gas analyser (RGA) that collected a part of the exhaust gas from the dc-GD discharge.

2. Experimental setup and procedure

The experimental set-up is shown in Figure 1. It consists on a cylindrical stainless steel vacuum vessel (plasma chamber), which acts as the grounded cathode, pumped out with a turbomolecular pump and backed by a rotary pump (base pressure of 10^{-5}

Pa). High purity (>99.999%) gases (H_2, N_2, Ar) are introduced into the chamber by an electronic flowmeter. Inside this primary SS vessel, a concentric liner is placed. It consists of a thin SS sheet covered by high purity cold rolled tungsten. A thermocoax wire is rolled in the external wall of the liner, to heat it. The wall temperature is measured with a thermocouple and controlled by a power supply.

The plasma chamber is connected with the analysis chamber through a small collimator, where a differential pumped residual gas analyser is placed. This chamber is pumped down with another turbomolecular pump at a base pressure lower than 10^{-7} Pa. The pressure in both chambers is measured with ionization gauges, and a capacitance manometer (0.1-100 Pa) is used. A total pressure of 2 Pa was used in all the discharges. All the experiments were carried out after extensive wall conditioning in order to assure the reproducibility of the experiments. It consists on a pure DC-GD Ar plasma for 30 minutes followed by 1 hour of wall baking at 200ºC. In addition, the differentially pumped mass spectrometry detection system provides a ultra-high vacuum environment with very low residual water contents for the measurements, trying to avoid the perturbation of the measurements as water RGA peaks overlap with those from ammonia in the mass to charge signals collected by the mas spectrometer. However in the experiments performed with alternative N_2-D_2 plasmas, as a result of the very low ammonia formation (comparable to the persistent residual water content) derived from the specific experimental procedure, this overlapping among the signals was unavoidable For the experiments with simultaneous irradiation by means of N_2-H_2-(He) plasmas, the generation of ammonia was much higher, thus producing related mass to charge signals (mainly 17 amu/e signal) that were generally higher by two orders of magnitude compared to the residual water signals (18 amu/e and 17 amu/e) thus avoiding the overlapping problems as the residual 17 amu/e signal related to water was 100 times lower before starting the ammonia formation.

For the latter experiments, absolute calibrations of the single RGA peaks (28, 2 and 17 amu) to N_2, H_2 and NH_3 partial pressures respectively were performed prior to the experiments. The ammonia formation yields were normalized to the injected nitrogen (NH_3/N_2^{inj}) and "depleted" ($NH_3/2\Delta N_2$) nitrogen in the plasma as well as to the nitrogen content stored on the walls by means of particle balance calculations.

2.1. Alternate D_2/N_2 GD plasmas on N/D saturated tungsten walls for mechanism investigation

To approach these plasma-surface chemistry studies two different experiments were carried out:

- The first one consisted on performing a pure D_2 plasma on the tungsten walls previously loaded with nitrogen after the exposure to a pure N_2 GD plasma. Nitrogen saturation of the W wall was achieved by running a pure N_2 GD plasma (p=2Pa, plasma current of 300 mA and discharge voltage of 400 V) up to a total fluence of 10^{23} N/m^2). After this, the N-saturated surface is irradiated with pure D_2 GD plasma (p= 2 Pa, total D/N fluence of 10^{23} m^{-2}, plasma current of 350 mA and discharge voltage of 500 V). During this irradiation the gas exhaust composition was monitored with the mass spectrometer. The differential pumped system assures low impurity (including water) content in the analysis chamber.
- The second one was the symmetric experiment, i.e., producing the saturation of the W wall with pure D_2 GD plasma followed by irradiation with pure N_2 GD plasma. The characteristics of the glow discharge plasmas and the wall temperature and conditioning procedure were the same as for the experiment previously commented.

For the both experiments, the following mass to charge ratio signals were monitored with the mass spectrometer: 2, 3 and 4 amu/e (H_2, HD and D_2), 15-20 amu/e (contributions from water and ammonia molecules), 28 amu/e (mainly nitrogen) and 30 amu/e (NO). The presence of deuterated and hydrogenated ammonia/water overlapping species (NH_3, NH_2D, NHD_2, ND_3, H_2O, HDO and D_2O) made the mass spectrometry analysis for the quantification quite complicated. To separate the contribution of the different species in the overlapping masses a deconvolution calculation of the signals was necessary, taking into account the cracking patterns for NH_3 and H_2O species.

2.2. Effect of helium addition in the ammonia formation in N_2-H_2 mixed plasmas

These experiments consisted on the irradiation of the tungsten wall of the plasma chamber with N_2-H_2 mixed plasmas. They were performed in two phases: a first part at constant plasma current of 1-1.5 hours of duration, until the steady state was reached in the system. Later, a second phase with scans in plasma current was

performed. The discharge characteristics during the experiments were: discharge voltage between 300-400 V, plasma current of 200-350 mA and hydrogen/nitrogen total fluences up to 10^{23} m^{-2}. Additionally, Optical Emission Spectroscopy measurements were performed to estimate the electron temperature (T_e) and its changes during the helium addition to the plasmas, measuring several He emission lines in the 400-750 nm wavelength range. The electron temperature of the plasma is an important factor for the ionization/dissociation (plasma-gas phase) processes that could change due to the presence of helium in the plasma, thus inducing a possible modification in the ammonia formation yields.

3. Experimental results

3.1. D_2 plasma on a W wall saturated with nitrogen

In figure 2, the evolution of the RGA signals during the experiment is displayed. During the first part of the experiment (from 5940 s to approximately 6000 s) D_2 is introduced in the plasma chamber with the W wall previously saturated by a N_2 GD plasma. Special care was taken to avoid a possible contamination with water or residual air during the gas injection in the system that might disturb the mass spectrometry analysis. For this purpose, in these experiments (and in the symmetric ones) the gas inlet lines were previously purged through the vacuum system, checking with the mass spectrometer the species that were injected during the puffing until assuring that only D_2 or N_2 are introduced in the chamber through the gas inlet flowmeters.

No changes in the signal evolution were observed during this gas injection. In the subsequent plasma phase a decrease in the 4 amu/e signal (due to the cracking of the deuterium molecules) and a concomitant increase in the 17-20 signals are clearly visible. The increase in the RGA signals at 17-20 amu/e reveals the formation of different ammonia molecules. They are discriminated in the mass spectra due to the different H/D content. In principle no hydrogen is injected in the system during the experiment, hence no hydrogen should be present in the ammonia molecules. Despite the high vacuum level present in the analysis chamber, the presence of small amounts of water molecules or residual hydrogen from impurities in the chamber or on the tungsten wall is unavoidable, thus providing the necessary hydrogen atoms to produce hydrogenated ammonia molecules. Moreover from the experiments with N_2 seeded discharges in JET tokamak [5], it is well-known that deuterated ammonia is prone to an intense isotope exchange with hydrogen that determines a high degree of replacement of

D atoms for H atoms during the transportation from the surfaces to the mass spectrometer sensors through the pumping lines.

These reasons may explain the presence of important amounts of hydrogenated ammonia in our experiment. During the D_2 plasma performance the formation of ND radicals (that acts as precursors for the ammonia formation) must be initiated on the wall because there is no presence of nitrogen in the gas phase (nitrogenated W wall exposed to pure D_2 GD plasma). The recombination reactions can happen by means of L-H recombination between the previously adsorbed nitrogen and adsorbed deuterium atoms from the plasma, or by means of E-R recombination between adsorbed nitrogen and impinging deuterium from the plasma. In this way, the chemical reactions related with the formation of ammonia take place on the tungsten surface. Hence, this experiment confirms the crucial role of the nitrogen legacy in the surface chemistry processes that determines the ammonia formation. The gas/plasma chemistry contribution in the ammonia formation cannot be totally rejected but at least in this experiment its role seems to be very limited compared to the surface processes due to the low pressure conditions.

For the quantification of the products in the range of 17-20 amu/e, deconvolution of the spectra is necessary as water and ammonia molecules with different H/D content are isotopologues and overlap in the amu/e signals. In the case of ammonia these contributing molecules are NH_3, NH_2D, NHD_2, and ND_3, while for the case of water they are H_2O, HDO and D_2O. As a result of the interaction with the RGA ionization chamber during their detection, the different ammonia and water molecules are cracked in several partially dissociated fragments that contribute to the global values of the 15-20 amu/e signals detected by the mass spectrometer. These fragments are formed by depleting one or more hydrogenic atoms from the neutral molecule. The relative intensities of the resulting peaks registered by the RGA, directly related with the expected proportion of these partially dissociated species in the measurement, are known as cracking patterns. By using these cracking patterns (experimentally determined in our case) it is possible to formulate a set of equations with the time-integrated values of the 15-19 amu/e signals. By considering as negligible the formation of the most deuterated ammonia molecule ND_3 (assumption favoured with the intense D/H isotope exchange of ammonia to hydrogen [5]) and then removing the 20 amu/e species from the system, the system is simplified to a set of 5 equations (15-19 amu/e signals) with 5 molecules (NH_3, NH_2D, NHD_2, H_2O and HDO), whose solution gives

the contribution of each molecule to the mixture. Unfortunately by applying this method the results obtained were not successful as the solution had not a physical sense. Probably the experimental data are not complete enough for the quantification due to the possible losses (ammonia sticking [6]) and condensation of a part of the ammonia and water species in the system before reaching the RGA and be detected. To avoid this problem and have a better analysis, a solution would be to use the cryo-trap assisted mass spectrometry technique (CTAMS, [7]) that uses a liquid nitrogen circuit for the condensation of all the ammonia and water molecules before they reach the RGA. It assures that all the molecules are trapped by the condensation and then finally evaporated and analysed in the RGA without any loss in cold parts of the system.

3.2. N_2 GD plasma on a D-saturated W wall.

The mass spectrum evolution over time for the experiment is shown if Figure 3. As in the previous and symmetric experiment, the first part of the spectrum corresponds with a gas injection (in this case nitrogen). During this gas feed a clear increase in the 18 amu/e and 19 amu/e signals can be observed, being the increase in the 20 amu/e smaller. No significant increase in 17 amu/e is detected. In the subsequent plasma phase these signals decrease until the background level before the injection of nitrogen and the 2 amu/e (H_2), 3 amu/e (HD) and the 30 amu/e (NO) RGA signals increase. No changes in the 17 and 20 amu/e signals are found during the plasma phase. This experimental observation can be interpreted in this way: during the N_2 gas injection there is a decrease in the pumping speed of the system due to the increasing pressure and the concomitant change in the pumping efficiency of the turbo-molecular pump. In this way the residual level of water molecules (that even could be affected by small contamination in the puffing lines) increases (mainly 18 and 19 amu/e signals). Due to the H/D isotope exchange reactions of this residual water on the deuterium saturated tungsten wall, amounts of partially (DHO) and totally (D_2O) deuterated water are produced. These molecules contribute also to the increase in the 19 and 20 amu/e signal that are visible in Figure 3. During the plasma phase these molecules of water are decomposed, producing the depleted hydrogen and deuterium the H_2 and HD molecules that increase 2 and 3 amu/e traces in the RGA spectrum. The oxygen depleted from the water molecules produces NO molecules due to interaction with the nitrogen active atoms that are being trapped on the tungsten surface. This NO production can be seen as an increase in the 30 amu/e signal during the plasma phase. Although there is no a direct

calibration of the water involved species, the comparison of the time integrated values of 18 amu/e and 19 amu/e increase during the gas phase and the subsequent integrated values for the increase of the mass traces of HD and H_2 during the plasma phase shows a ratio around 0.9. This result also points to the water molecules cracking during the N_2 plasma and their conversion into HD and H_2. This calculation together with the formation of NO that happens in the system during the plasma phase seems to corroborate our assumption about the water contamination and shows qualitatively that no ammonia is significantly produced in this symmetric experiment. Hence, once again, it is demonstrated that the presence of dissociated nitrogen on the W surface is the first mandatory step necessary to trigger the ammonia formation process. The previously implanted deuterium atoms from the first plasma have a larger implantation range compared to the nitrogen ones. According to this premise, the nitrogen atoms depleted from the plasma could not significantly interact with the deuterium atoms present (deeper) on the W surface, thus minimizing the formation of ammonia.

3.3. Enhanced N-H recombination induced by the helium bombardment

In Figure 4 the ammonia formation yields is presented for the case of N_2-H_2 plasma and three different N_2-H_2-He plasmas (He contents ≈ 1.5, 3 and 8 %).

Both yields are enhanced up to a 45% factor for increasing helium plasma content by comparing values with similar nitrogen cracking efficiency (parameter that represents the fraction of injected nitrogen that disappears in the plasma). Possible changes in the electron temperature (T_e) of the plasma that could lead to an improved dissociation of the nitrogen/ammonia molecules thus determining increased formation yields were examined. By using the He emission line ratios and the electron temperature dependence of the excitation cross sections of the associated transitions as well as the transition probabilities, the T_e value can be calculated. The results shown electron temperatures around 3±2 eV [8], but the limited accuracy of the measurements cannot conclude that the T_e parameter is increased for higher helium plasma contents, hypothesis that would explain (at least partially [8]) the enhanced ammonia formation observed. Furthermore, the experimental data showed a very similar N_2 cracking efficiency for plasmas with identical current but different helium plasma contents, thus pointing to negligible effects of the helium presence in the electron temperature and associated nitrogen/ammonia dissociation. Additionally, a gas test was also performed to check the influence of the helium presence in the RGA detection (induced by changes

in the pumping efficiency of the system due to the helium injection) of the ammonia that could disturb its quantification, showing that the amplification effect in the detection is quite smaller compared to the observed enhancement in the ammonia formation [8].

Consequently, the changes observed in the ammonia production must be related with the modification of the surface chemistry produced by the tungsten bombardment with energetic helium (E ~ 400 eV). The ammonia molecules are produced by successive N-H recombination reactions between the nitrogen atoms adsorbed on the catalytic centres of the tungsten surface with impinging (E-R) or adsorbed (L-H) hydrogen neutrals [9]. In principle, the impact of such ions on the surface could modify the tungsten matrix morphology, thus affecting to the amount of catalytic active sites where the recombination takes place. Formation of filaments and/or an increase in the roughness derived from this bombardment will be also translated in a higher recombination area for the studied reactions. The activation of additional surface sites might also take place on the tungsten substrate as a consequence of the previously commented factors. All these effects will be translated into an enhanced N-H recombination that would increase the ammonia formation as was observed in the experiments. The formation of nanostructures denominated as “fuzzy tungsten” is a well-known phenomenon that takes place when this material is bombarded with energetic helium. Although the irradiation conditions for such formation are more severe in terms of total fluence, energetic content and surface temperature [10] (thus probably precluding the creation of fuzzy structures), another modifications in the basic morphology could be produced. To explore it, the complete characterization of the surface state in future experiments, (employing *in situ* measurements or post mortem techniques) would be of paramount importance.

4. Conclusions

Despite the simplicity of the experiments and the impossibility of accurate quantification, the alternative N_2/H_2 plasma results are useful for a qualitative and phenomenological characterization of the involved surface chemistry. In the first experiment, the previous presence of nitrogen (legacy) leads to the formation of ammonia when the wall is bombarded with D_2 plasma. On the contrary when nitrogen is not present on the wall previously, ammonia is not formed, thus confirming that the

limiting steps in the reaction are the dissociation and the adsorption of N atoms on the active sites of the tungsten surface.

Although the nitrogen plasma of the second experiment produces the dissociation of nitrogen and thus, the N atoms can be adsorbed on the wall and react with the deuterium ones present on the wall through N-H recombination, this does not happen. For an L-H recombination the adsorption of N and H species must take place in determined catalytic active centres of the surface with sufficient energy to recombine. However it is well-known that the implantation range of deuterium on tungsten is significantly larger compared to nitrogen. Hence as a consequence of the experimental procedure the deuterium plasma loading will accumulate the D atoms in positions deeper compared to the posteriorly loaded nitrogen ones, thus precluding the mutual interaction of N and D and the formation of ammonia. Diffusion into the bulk of the previously loaded deuterium or the insufficient energetic content of the deuterium present on the wall can also preclude the effective recombination. However the first experiment shows that the saturation of the catalytic centres with nitrogen does not preclude the ammonia formation process. The subsequent impinging deuterium particles can react via E-R recombination or by means of L-H recombination as the impinging deuterium atoms can interact with the previously loaded nitrogen (implantation range of nitrogen smaller than deuterium). Unfortunately, the RGA measurements can only the characterize the stable reaction products, being impossible to differentiate if both mechanisms are taking place on the W wall at the same time. To perform this investigation and study in a broader way the chemical mechanism of the ammonia formation, complementary techniques that involve the *in situ* diagnosis of the wall state with direct measurements that can detect the presence of adsorbed species and radicals would be necessary.

The surface modification induced by the helium bombardment seems to play a key role that increases the net ammonia formation. The structural deformation on the tungsten surface induced by the helium bombardment could alter the roughness (thus affecting the effective recombination area) and the total number of free centres available for recombination even affecting the activation of the catalytic sites for N-H recombination (plasma activation). The conjunction of these surface effects could explain the observed results.

To understand these effects, a further investigation (involving the in situ diagnosis of the W matrix [LIBS] or post-mortem characterization of the surface as XPS, ellipsometry, SIMS…) would be necessary in future works, being the employment of

surface analysis techniques completely necessary for a better comprehension and quantification of the involved surface chemistry parameters. The present results provide an approximation to the complex chemistry that determines the ammonia formation in a N_2-seeded plasma operation but is necessary to keep in mind that their extrapolation to the conditions of a hot and fully ionized plasma with much higher particle fluxes existing in the divertor of a fusion reactor may be not straight forward. In this sense, it is important to note that another experiments performed in linear plasma devices and aimed to investigate the influence of helium in the ammonia formation process have obtained opposite results about the effects of the helium presence in the ammonia production yield [11, 12]. An important difference between our plasmas and linear device/divertor plasmas is that the energetic content of the helium ions bombarding the surface is higher, while the particle flux is quite lower. The magnitude of the involved particle fluxes and the energy content of the species seems crucial to compare among these experiments performed under different experimental conditions.

5. Acknowledgments

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Figure captions

Figure 1. Experimental Setup. 1. Pumping system, 2. Manometer (Bayard Alpert), 3. Capacitance manometer, 4. Diaphragm (Differential pumping), 5.Anode, 6. Concentric SS liner, 7. Gas inlet, 8. Optical window, 9. Mass spectrometer, 10. Electron gun

Figure 2. Pure D_2 GD plasma on a W wall previously saturated with a pure N_2 GD plasma

Figure 3. N_2 GD plasma on a W wall previously saturated with a D_2 GD plasma

Figure 4. Influence of the helium plasma content in the ammonia formation yields

Figure 1.

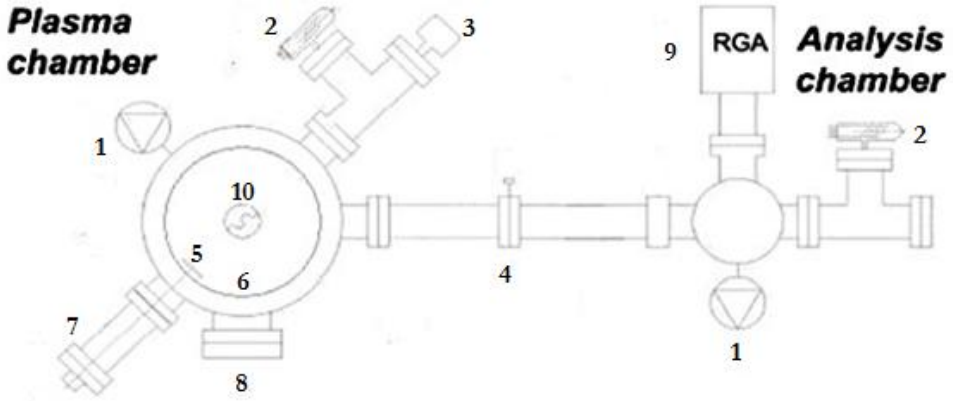

Figure 2.

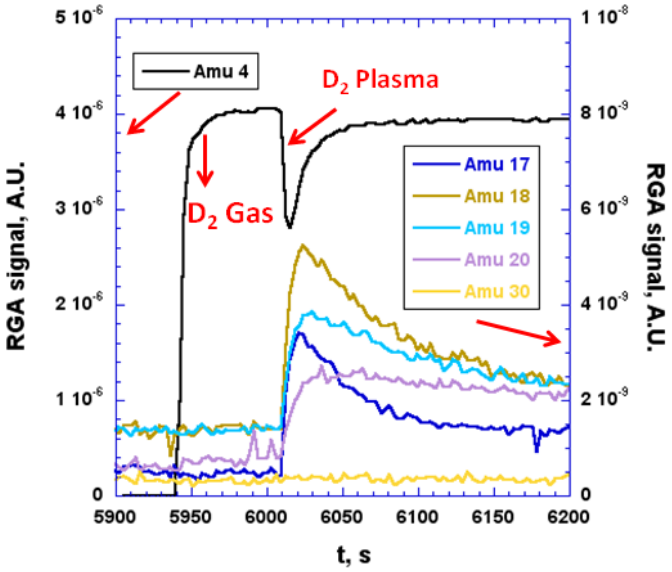
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Figure 3.

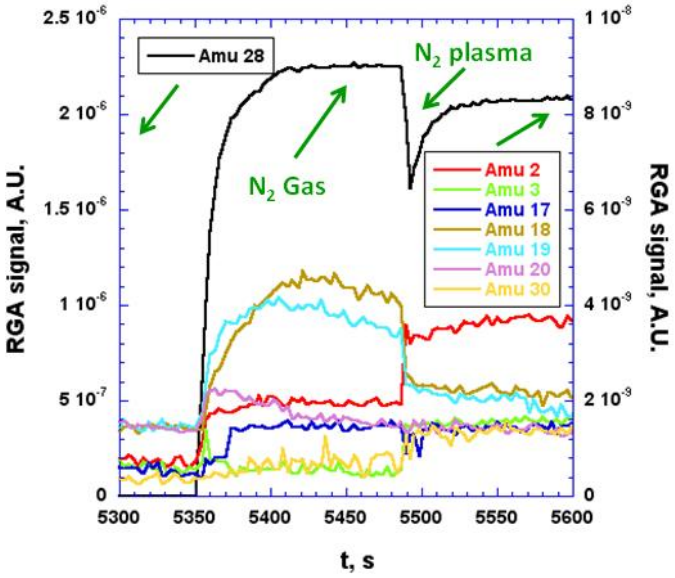
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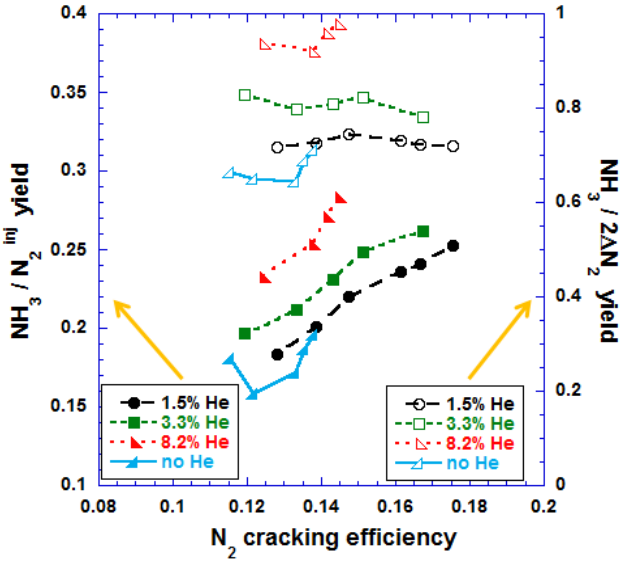
Figure 4.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NH3 / N2 inj yield
NH3 / 2ΔN2 yield
N2 cracking efficiency
1.5% He
3.3% He
8.2% He
no He
1.5% He
3.3% He
8.2% He
no He