

Characterisation of a novel plasma system for application in ultra-precision processes

Katherine Gobey¹, Dr Adam Bennett², Professor Chris Sansom³, Dr Peter King⁴

¹Doctoral Researcher, Cranfield University, Bedford, Bedfordshire, MK43 0AL, UK

²Director, Cranfield Plasma Solutions, Brooks Close, Burton Latimer, NN15 5PX, UK

³Professor, Renewable Energy Systems Centre, Cranfield University, Bedford, Bedfordshire, MK43 0AL, UK

⁴Research Fellow, Renewable Energy Systems Centre, Cranfield University, Bedford, Bedfordshire, MK43 0AL, UK

Katherine.j.gobey@cranfield.ac.uk

Abstract

The use of Atmospheric Pressure Plasma (APP) holds much promise for processing without the high cost and consumption of time of traditionally used vacuum plasmas, however operation at atmospheric pressure necessitates generation of a plasma that is stable spatially and temporally, for processing that is controllable and repeatable. This work employs different methods of characterisation to investigate stability and distribution of plasma generated by an APP device.

Atmospheric Pressure Plasma (APP), spectroscopy, X-ray computed tomography (XCT), precision manufacture, metrology, in process monitoring,

1. Introduction

Many precision processes such as during the manufacture of films, application of coatings, and adhesion of polymers, require a surface clean and free of contamination and soiling [1] [2] [3]. Plasma has been utilised extensively for cleaning of the surface however occurs most commonly in vacuum plasma systems that are time consuming and costly [4]. Atmospheric pressure plasmas (APP) are an area of increasing interest in research as they can enable processing of materials in-process, without the need for vacuum chambers [5]. As plasmas generated at atmospheric pressure do not exist uniformly as in a vacuum chamber, characterisation of APP is important to ensure repeatability and optimisation of processing [6].

Presented here is characterisation of a novel plasma generation device from Adtec Plasma Technology that utilises an array of plasma to allow faster treatment of larger areas.

2. Background

There is huge variation in plasmas produced both within industry and research, indicating the wide variety of applications plasma has. Sufficient determination and control of these characteristics are vital for plasma's integration in industrial processes, as poorly optimised or controlled parameters can result in damaged materials, inconsistencies in production, or inadequate processing effects. Plasma temperature is one such variable that can have considerable effect on processing capabilities [7]. Due to high energies required for ionisation, plasma temperature is usually high, in some cases up to 10000 K [8]; however it is possible to create "cool" plasmas, which are used for more delicate applications such as in medical applications, for example wound sterilisation [9], treatment of polymers [10] [11], and processing of crystal quartz [12].

In atmospheric pressure plasmas distribution of species within the plasma jet or array is also of considerable importance. In particular plasmas utilising reactive or multiple species require precise spatial control to ensure processing effects are only on the desired areas. Careful control of gas flow both within and upon exit of the torch is often necessary to ensure the plasma is contained in a repeatable manner [13].

3. Methodology

Three methods of characterisation were undertaken: X-ray computed tomography (XCT), for a better understanding of the structure of the torch itself; spectroscopy in the UV-Visible-Near Infrared range to characterise the plasma produced; and measurements of torch temperature, to determine attainment of thermal equilibrium. These were then compared to better understand the data obtained from each.

3.1. X-ray computed tomography (XCT)

For this the torch was placed on a motion stage within the XCT chamber, as shown in Figure 1, with parameters as shown in Table 1, and images generated at various angles and positions such that an image of the internal composition of the torch was built up. This work was carried out at the National Physical Laboratory (NPL), London.

Beam Energy	170 kV
Beam Current	80 μ A
Beam Power	13.6 W

Table 1. Parameters of beam used for XCT analysis.



Figure 1. Experimental setup for XCT of torch

3.2. UV-Visible-Near Infrared Spectroscopy

The second method of characterisation was using UV-Vis-NIR spectroscopy. The torch was mounted in an enclosure below which a lens was positioned on a motion stage (Figure 2). Spectrometers (OceanOptics) were used to obtain emission spectra from the plasma for both spatial mapping, where the lens travelled to cover an area across the plasma, and temporal mapping, where the lens was stationary under each electrode.

A static whole spectrum (200 nm – 850 nm) was taken first to determine the composition of the plasma, from which a single peak was chosen for analysis of temporal and spatial stability. Plasma was generated first using a supply of clean dry air (CDA), then with pure nitrogen gas, with gas flow rate and forward power kept constant.

3.3. Temperature measurements

Measurements of temperature were taken from a temperature sensor built into the device by Adtec Plasma Technology. Readings were taken every 30 seconds from a start temperature of 25 °C up to 80 °C, at which a safety limit is reached and system shuts down. This was done with and without application of a rudimentary cooling system (a flow of clean dry air over the upper section of the torch).

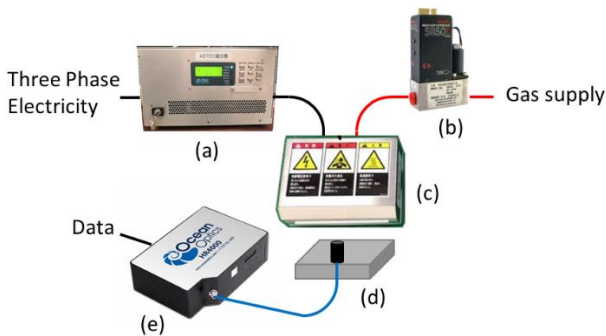


Figure 2. Schematic for attainment of spectroscopy data: (a) RF Generator, (b) Mass Flow Controller, (c) plasma generating device, (d) lens mounted on motion stage, (e) Spectrometer.

4. Results and Discussion

4.1. X-ray computed tomography (XCT)

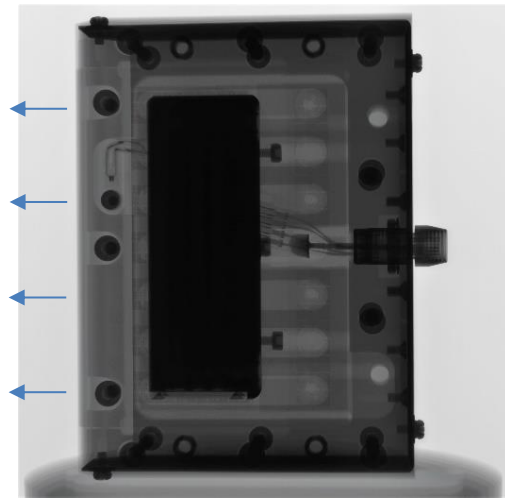


Figure 3. XCT image of plasma generating device, with direction of plasma generation indicated with arrows.

The image obtained using XCT (Figure 3) showed the torch unit comprising of an array of micro-discharges arranged into seven electrodes. Also observed is the temperature sensor and associated connections.

4.2. Air plasma

Plasma generated from the CDA supply exhibited maximum intensity at the 337.13 nm peak which corresponds to the second positive system emission of nitrogen [14] (Figure 4). The majority of peaks observed can be attributed to emission from nitrogen, with the exception of a small peak at 272.2 nm which occurs from the nitrogen third positive system of Nitric Oxide (NO) [14]. Therefore it can be concluded that the plasma generated here is almost entirely nitrogen plasma, with negligible ionisation of other components in the air such as oxygen and carbon dioxide. The relatively low intensities of nitrogen observed suggest considerable energy is wasted on these species providing thermal kinetic energy at a level below which ionisation occurs.

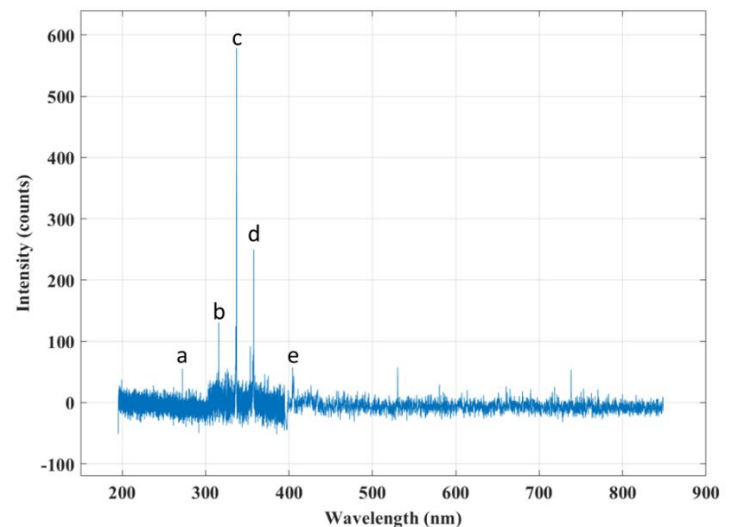


Figure 4. Emission spectrum of air plasma with peaks labelled as follows: a, NO A-X; b, N₂ C-B (1-0); c, N₂ C-B (0-0); d, N₂ C-B (0-1); and e, N₂ C-B (0-3) [15].

As a result of the low intensities of emission, noise from the spectrometer presented a considerable challenge in accurate analysis of data, consequently this has been omitted from further discussion presented here.

4.3. Nitrogen plasma

Emission spectrum in the whole measured range from the plasma generated using pure nitrogen demonstrated the presence of nitrogen plasma (Figure 5), with the most prominent peak observed at the same 337.13 nm nitrogen peak as in the air plasma. This peak was used for all further spectroscopy analysis. Observed intensities were considerably higher than for air plasma, suggesting a far higher degree of ionisation and therefore more efficient plasma generation.

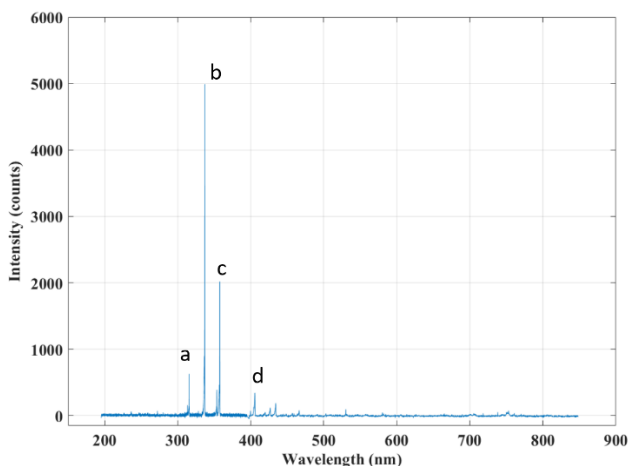


Figure 5. Emission spectrum of Nitrogen plasma with peaks labelled as follows: a, N₂ C-B (1-0); b, N₂ C-B (0-0); c, N₂ C-B (0-1); and d, N₂ C-B (0-3) [15].

Spatial mapping observed 7 distinct peaks in emission intensity at the 337.13 nm peak (Figure 6), with a roughly Gaussian distribution in both x and y. When compared to the image obtained via XCT, it can be concluded that the plasma generating device consists of 7 distinct electrodes (Figure 7).

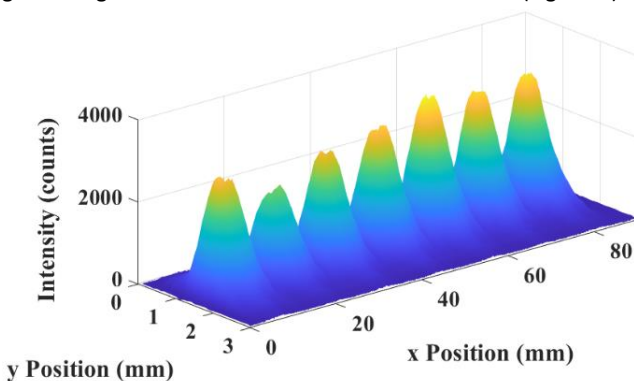


Figure 6. Intensity of 337.13nm peak of nitrogen plasma in x and y.

Spatial mapping also showed a significantly lower emission at the second electrode. This corresponds with the location of the temperature sensor as shown in the XCT image, suggesting the placement of this has interfered with the plasma during or after generation.

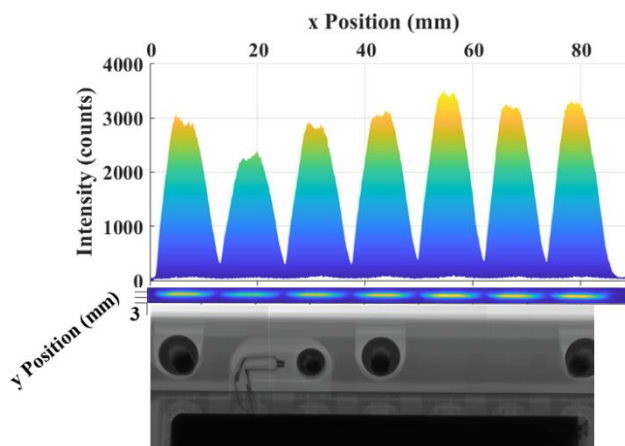


Figure 7. Intensity of 337.13nm peak relative to x position of the plasma emitting device (above), and intensity of peak at x and y position (middle), aligned with electrodes visible in XCT image (below).

Temporal stability measurements (as shown in Figure 8) showed the nitrogen plasma discharged from each electrode to be temporally stable to within $\pm 6\%$, with a gradual increase in intensity. It also shows each electrode experiences a brief (approximately 15 seconds) ramp up of intensity before becoming more stable. A gradual increase in intensity is observed over the time elapsed, suggesting the plasma did not reach thermal equilibrium during the duration of data collection. This may also explain the seemingly large envelope of instability.

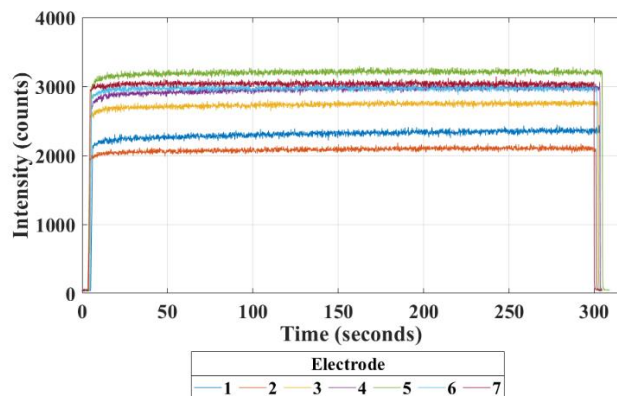


Figure 8. Intensity of 337.13nm peak relative to time for each of the electrodes.

Also apparent from temporal stability data is disparity in the average intensity emitted at each electrode. Average intensity for all electrodes was 2757 counts, with the highest average being electrode 5 at 3203 counts, and the lowest average of 2084 counts at electrode 2. This considerable range in intensities may be due to initial prototype design of the plasma generating device and/or lack of precision in manufacture of the torch electrodes.

Temperature measurements (Figure 9) demonstrated the need for cooling, which significantly increased running time despite its basic nature. It also increases the possibility of reaching thermal equilibrium as seen by the heating curve levelling off. It is possible that if left to run this equilibrium of the Nitrogen plasma would have been reached, however gas use constraints meant it was necessary to switch off the plasma torch before this could be observed. Nitrogen plasma with cooling heated at a far slower rate than the air plasma, which may be due to the higher incidence of ionisation (and therefore more efficient generation if plasma) than in the air plasma, where significant energy went into heating molecules that were not subsequently ionised. Cooling of the torch without

application of external cooling air flow was observed to approximately obey Newton's law of cooling.

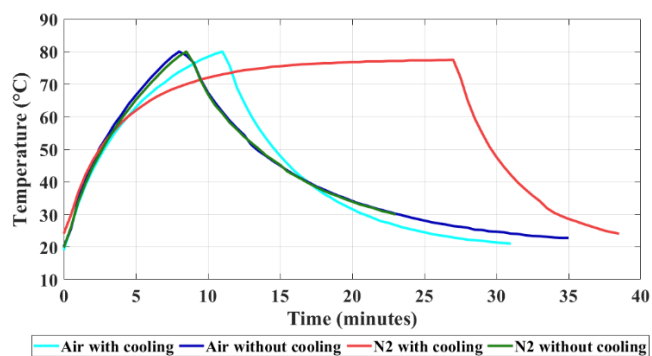


Figure 9. Plasma generating device temperature with and without cooling.

5. Conclusion

Investigation of the plasma generated by the device has shown the importance of torch design and construction for the generation of a stable plasma. Also demonstrated is the importance of characterisation of plasma produced, such that processes and/or torch design may be altered to ensure any processing conducted using the device is stable, repeatable, and optimised. Additionally, it has been shown that it can take considerable time for the torch to reach thermal equilibrium, in this instance greater than 5 minutes, resulting in a plasma that is temporally unstable. Thus, time until equilibrium must be determined and taken into account for processing applications. An efficient cooling system is can help achieve this, and allows for longer running times that are beneficial for industrial applications.

6. Future Work

Factors that affect plasma stability are numerous and will vary between systems. Typically, these include physical factors such as torch design, as well as more easily variable parameters such as gas flow and forward power. Future work will be conducted to optimise these parameters to produce a more stable plasma than was investigated here. Additionally, further analysis of spectroscopy data will enable a better understanding of distribution of species within the plasma.

The effect of stability and distribution of plasma on control and repeatability of processing, such as surface energy modification, cleaning, and deposition of coatings at atmospheric pressure, will be explored.

Acknowledgements

This work was supported by the UK EPSRC under grant EP/K503241/1 (Centre for Doctoral Training in Ultra Precision Engineering) and EP/L016389/1 (Centre for Doctoral Training in Sustainable Materials and Manufacturing). The author would also like to thank the National Physical Laboratory (NPL) for use of their XCT equipment; ADTEC Plasma Technology & ADTEC Europe for providing financial and technical support and bespoke plasma equipment; and Cranfield Plasma Solutions for technical advice and knowledge.

References

1. Inoue S., Takenaka S., Shimoda T. Study of degradation

phenomenon due to a combination of contamination and self-heating in poly-Si thin film transistors fabricated by a low-temperature process. *Japanese Journal of Applied Physics, Part 1: Regular Papers and Short Notes and Review Papers*. 2003; 42(7 A): 4213–4217. Available at: DOI:10.1143/jjap.42.4213

2. Gray JE., Luan B. Protective coatings on magnesium and its alloys - A critical review. *Journal of Alloys and Compounds*. 2002; 336(1–2): 88–113. Available at: DOI:10.1016/S0925-8388(01)01899-0
3. Liston EM., Martinu L., Wertheimer MR. Plasma surface modification of polymers for improved adhesion: A critical review. *Journal of Adhesion Science and Technology*. 1993; 7(10): 1091–1127. Available at: DOI:10.1163/156856193X00600
4. Aronsson BO., Lausmaa J., Kasemo B. Glow discharge plasma treatment for surface cleaning and modification of metallic biomaterials. *Journal of Biomedical Materials Research*. 1997; 35(1): 49–73. Available at: DOI:10.1002/(SICI)1097-4636(199704)35:1<49::AID-JBMM>3.0.CO;2-M
5. Schütze A., Jeong JY., Babayan SE., Park J., Selwyn GS., Hicks RF. The atmospheric-pressure plasma jet: A review and comparison to other plasma sources. *IEEE Transactions on Plasma Science*. 1998; 26(6): 1685–1694. Available at: DOI:10.1109/27.747887
6. Bennett A. Microwave generated reactive plasma for ultra-precision technologies. 27th International Symposium on Plasma Physics & Technology. Prague; 2016.
7. Malyshev M V., Donnelly VM., Downey SW., Colonell JI., Layadi N. Diagnostic studies of aluminum etching in an inductively coupled plasma system: Determination of electron temperatures and connections to plasma-induced damage. *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films*. 2000; 18(3): 849–859. Available at: DOI:10.1116/1.582266
8. Yu N. Thermal Analysis of Energy Beam using De-Laval Nozzle in Plasma Figuring Process. 2016.
9. Arndt S., Unger P., Berneburg M., Bosserhoff AK., Karrer S. Cold atmospheric plasma (CAP) activates angiogenesis-related molecules in skin keratinocytes, fibroblasts and endothelial cells and improves wound angiogenesis in an autocrine and paracrine mode. *Journal of Dermatological Science*. 2018; 89(2): 181–190. Available at: DOI:10.1016/j.jdermsci.2017.11.008
10. Yuji T., Urayama T., Fujii S., Mungkung N., Akatsuka H. Temperature behavior of atmospheric-pressure non-equilibrium microwave discharge plasma jets for poly(ethylene naphtharate)-surface processing. *Surface and Coatings Technology*. Elsevier B.V.; 2008; 202(22–23): 5289–5292. Available at: DOI:10.1016/j.surfcoat.2008.06.056
11. Inagaki N., Narushima K., Morita M. Plasma surface modification of poly(phenylene sulfide) films for copper metallization. *Journal of Adhesion Science and Technology*. 2006; 20(9): 917–938. Available at: DOI:10.1163/15685610677657797
12. Bennett A. Processing of crystal quartz using atmospheric pressure microwave plasma technology. *European Society for Precision Engineering & Nanotechnology 18th International Conference*. Venice; 2018.
13. Vardelle A., Fauchais P., Dussoubs B., Themelis NJ. Heat generation and particle injection in a thermal plasma torch. *Plasma Chemistry and Plasma Processing*. 1998; 18(4): 551–574. Available at: DOI:10.1023/A:1021815417648
14. Pearse RWB., Gaydon AG. *The Identification of Molecular Spectra*. 1963.
15. Machala Z., Janda M., Hensel K., Jedlovský I., Leštinská L., Foltin V., et al. Emission spectroscopy of atmospheric pressure plasmas for bio-medical and environmental applications. *Journal of Molecular Spectroscopy*. 2007; 243(2): 194–201. Available at: DOI:10.1016/j.jms.2007.03.001