Bartosz HRYCAK, Dariusz CZYLKOWSKI, Robert MIOTK, Mariusz JASIŃSKI

Institute of Fluid Flow Machinery, Polish Academy of Sciences

doi:10.15199/48.2018.07.22

Spectroscopic characterization of plasma generated by microwave device for surface treatment

***Abstract**. In this paper, results of optical emission spectroscopy OES study of plasma generated in waveguide-supplied plasma-sheet microwave (2.45 GHz) plasma source (MPS)are presented. The plasma gas temperature inferred from rotational temperature of heavy species (assumed to be close to gas temperature) ranged from 800 up to 1300 K and the electron number density ranged from 3.3 * 10^{14} up to 6.1 * 10^{14} cm^{-3}. Moderate plasma gas temperature as well as high electron density makes presented plasma device attractive tool for different surface treatment.*

Streszczenie.** W tej pracy prezentujemy wyniki spektroskopowych badań plazmy wytwarzanej przez mikrofalowe (2,45 GHz), zasilane falowodowo, źródło płaszczyzny plazmowej. Temperatura cząstek ciężkich gazu (zakłada się, że jest ona bliska temperaturze gazu) wynosiła od 800 do 1300K natomiast koncentracja elektronów wahała się od 3.3 * 10^{14} do 6.1 * 10^{14} cm^{-3}. Umiarkowana temperatura gazu oraz wysoka koncentracja elektronów czyni z prezentowanego urządzenia atrakcyjne narzędzie obróbki różnorakich powierzchni.* ***Spektroskopowe badania plazmy wytwarzanej przez mikrofalowe zasilane falowodowo, źródło płaszczyzny plazmowej.

Keywords: microwave plasma, plasma sheet, optical emission spectroscopy (OES).
Słowa kluczowe: plazma mikrofalowa, płaszczyzna plazmowa, emisyjna spektroskopia optyczna (OES).

Introduction

Nowadays industry is highly interested in various surface treatment. Plasma surface treatment methods include processes like: cleaning, activating, etching the surface. These processes are used as pre-treatment of metal and plastic surfaces for further processes such as: soldering, gluing, painting, print. In contrast to chemical methods, the plasma methods do not require the chemical agents (solvents, acids, alkalis) and large amounts of water, so they are friendly to environment [1-3]. Plasma surface treatment methods also include such processes as: surface modification, surface coating, and thin film deposition. Using plasma methods allows to significantly change the properties of only the surface of materials without changing their properties at the greater depths. Plasma methods can be used for the processing of metals, polymers, glass and fibres, both natural and synthetic. They are used to change the surface properties such as wettability, adhesion, hardness, scratch resistance, permeability, corrosion resistance and others. [4-7]. Plasma surface treatment methods are used in the electronics, automotive, aerospace, textiles and biomedical industries (e.g.: implants) [8-11].

As a response to demand of industry recently we presented a novel compact microwave (2.45 GHz) plasma device for surface treatment [12, 13]. However the knowledge about plasma properties is necessary for development of plasma surface treatment technology. Optical emission spectroscopy (OES) is a powerful and useful tool in the characterization of plasma [14].

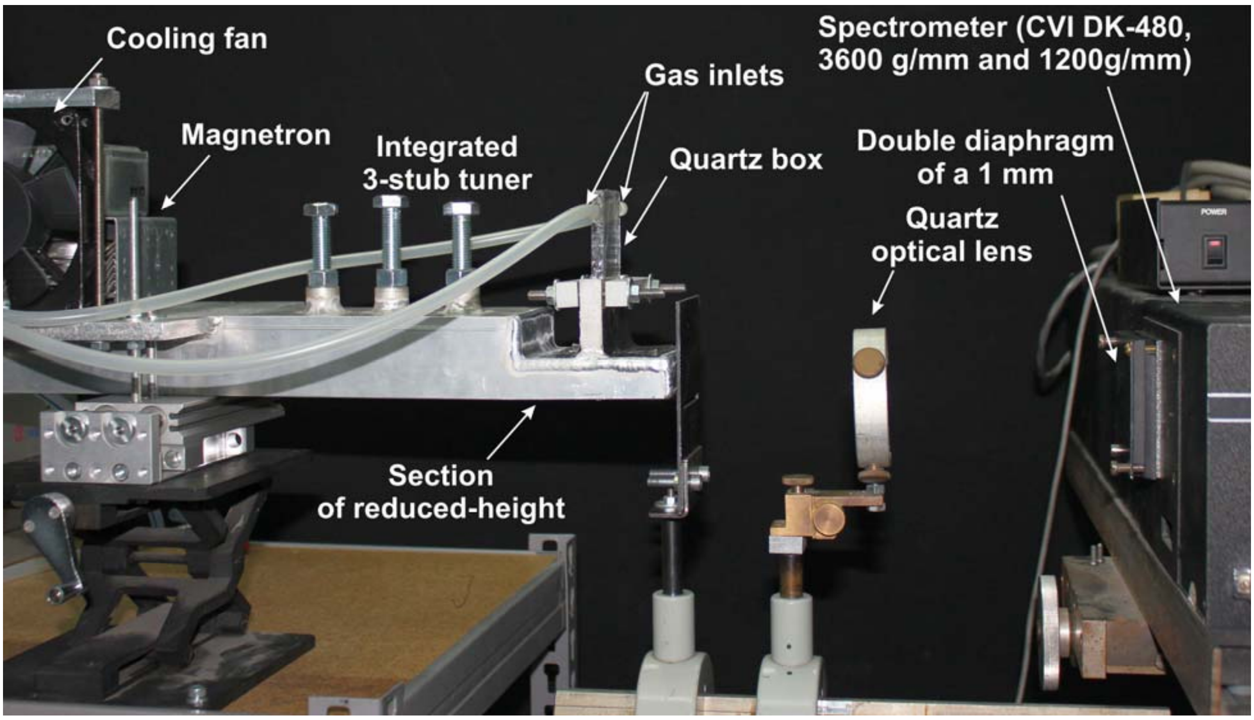

Fig. 1. The experimental setup for spectroscopic study of argon microwave atmospheric pressure plasma-sheet.

Experiments

The plasma was generated in waveguide-supplied microwave plasma-sheet source (MPS) operated at 2.45 GHz. Investigated MPS with integrated 3-stub tuner was based on WR 340 standard rectangular waveguide. An argon plasma was generated at the bottom of quartz box, which was inserted into the section of reduced-height of the waveguide, (see figure 1). The gas (argon) was introduced into the plasma by two ducts placed in quartz box's top. Standard magnetron type 2M240 was used as a microwave source. The magnetron was powered from Dipolar Magdrive-1000 industrial power supply. The atmospheric pressure argon at flow rate of 10-30 l/min was used as a working gas and the absorbed microwave power ranged from 300 to 850 W. The experimental setup presented in figure 1 consisted of: MPS, optical lens and spectrometer [DK-480 (CVI), 1200 and 3600 gr/mm grating] with sensitivity calibrated CCD camera and PC computer for spectra analysis. A small amount of pure hydrogen (up to 1%) was added optionally to gas flow in order to achieve measurable intensity of H_β spectral line. The emission spectra at the range of 300 - 600 nm were recorded. The rotational temperatures of OH radicals (assumed to be close to the gas temperature [15, 16]) were determined by comparing the measured and simulated spectra in LIFEBASE [17, 18] program. The electron number density in plasma was determined using the Stark broadening ($\Delta\lambda_S$) of H_β (486.13 nm) spectral line of the hydrogen Balmer series with formula [19]:

$$n_e = 10^{16}\left[(\Delta\lambda_S\,(H_\beta\;)^{1.55}\right]\,[\mathrm{cm^{-3}}] \quad (1)$$

The detailed procedures of temperature and electron number densities estimations were described by us elsewhere [20, 21].

The OH (A-X) rotational bands were measured using the 3600 grooves/mm grating and the all other spectra were recorded using the 1200 grooves/mm grating. Using a Hg-Ne low-pressure calibration lamp (λ = 435.84 nm, Hg I) we measured that in this experiment the Gaussian instrumental line profile FWHM was about 0.2 nm and 0.07 nm for 1200 and 3600 grooves/mm grating, respectively.

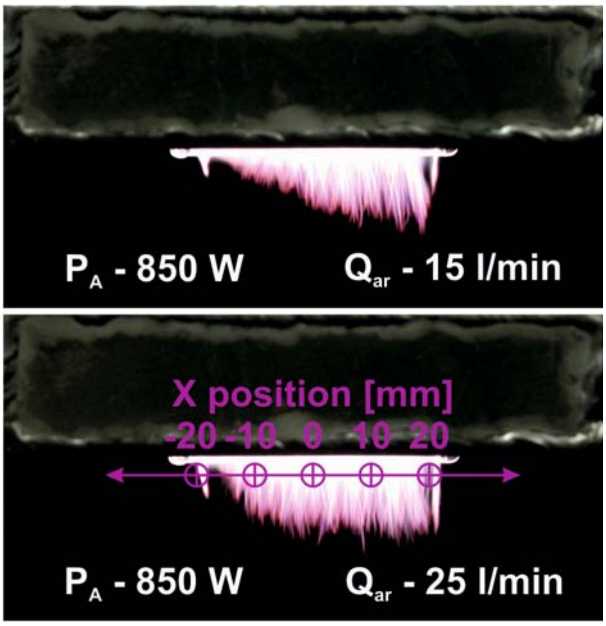

Fig. 2. Microwave plasma-sheet

Results

Figure 2 present the microwave plasma-sheet at two different argon flow rates 15 and 25 l/min. In the bottom photo the X axis and the positions of measurements are marked.

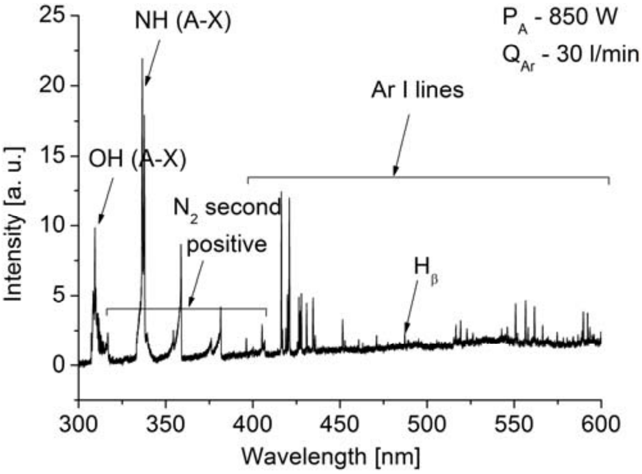

Fig. 3. Measured emission spectrum of argon plasma (P_A – 850 W, Ar flow rate Q_{Ar} - 30 l/min, X position - 0mm)

)

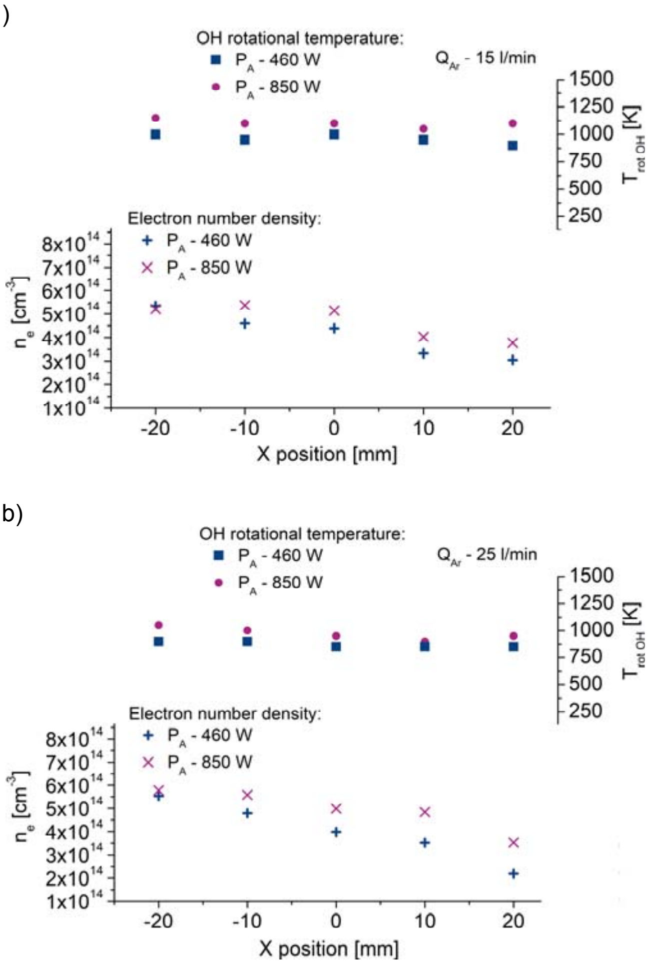

Fig.4. Rotational temperatures of OH radicals and electron number density as a function of X position in plasma for microwave absorbed power P_A 460 and 850 W and Ar flow rate Q_{Ar} 15 l/min

As it can be seen in figure 3 the dominant spectrum observed in the emission spectra of investigated plasma was NH (A-X) system. The spectra contained also N_2 second positive system (C-B) and OH (A-X) band. Presence of those bands indicated effect of strong entrainment of ambient air into the plasma. Thus in argon plasma we could observe also a Ar I lines in range above 400 nm. A low intensity H_β line could be also find in the spectra. A small amount of hydrogen up to 1% was added to the working gas in order to increase the intensity for H_β measurements. The addition did not influence significantly on plasma shape and spectra. On the other hand this addition increased intensity and allowed to separate H_β line profile from background noise with satisfying quality.

The distributions of the rotational temperatures of OH radicals and the electron number density in the plasma at argon flow rate 15l/min (a) and 25 l/min (b) for absorbed microwave powers of 460 and 850 W are presented in figure 4. The temperature of heavy species at this condition fluctuated at about 900 - 1000 K and electron number density ranged about $4*10^{14}$ - $5*10^{14}$ cm^{-3}. As it can be observed in figures 2 and 4 the plasma-sheet had an asymmetric profile as well as temperature and electron number density distribution. This asymmetry was probably caused by imprecise fabricating of the quartz box and further with asymmetric gas flow inside of it.

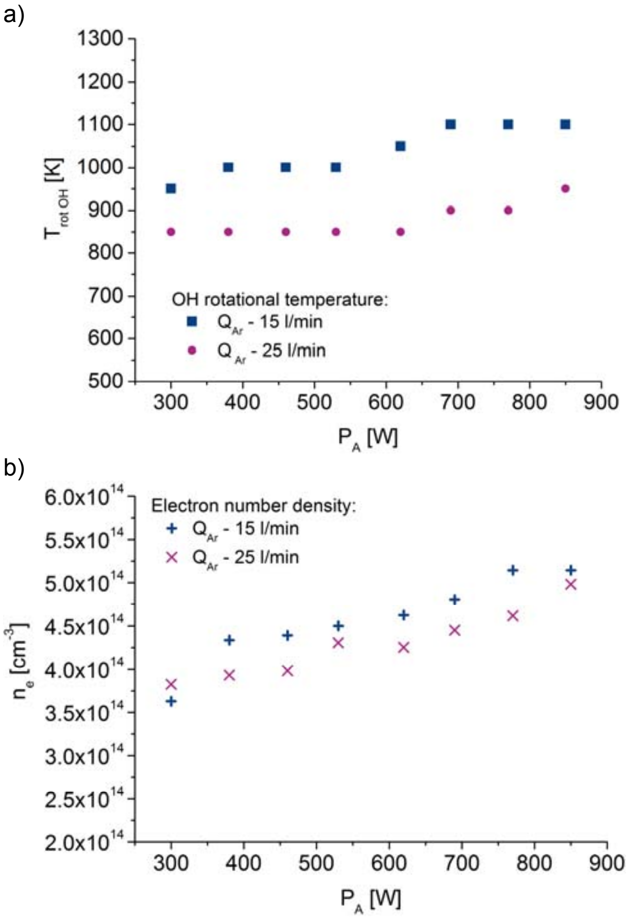

Fig.5. Rotational temperatures of OH radicals (a) and electron number density (b) as a function of microwave absorbed power P_A (for Ar flow rate Q_{Ar} - 15 and 25 l/min).

As it could be expected the rotational temperatures of OH radicals (see figure 5a) and the electron number density (figure 5b) increase with increasing microwave absorbed power. At the flow rate of 10 l/min and the absorbed microwave power of 850 W at the "hotter" side of the plasma (at X position = -20mm) the rotational temperatures of OH radicals exceeded 1300 K. At the same discharge conditions and the same location in plasma the highest measured electron density reached $6.1 * 10^{14}$ cm^{-3}.

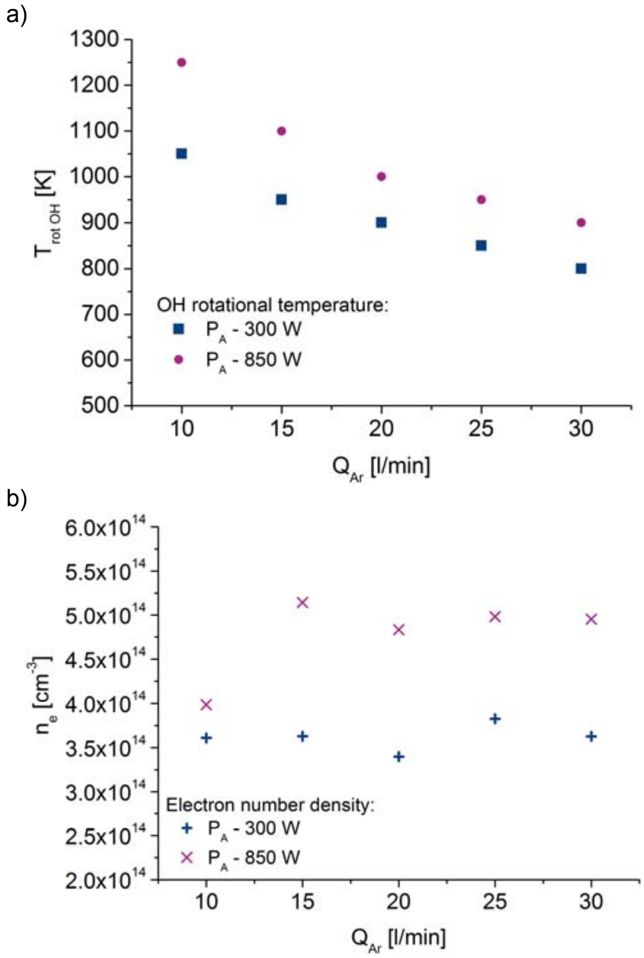

Fig.6. Rotational temperatures of OH radicals (a) and electron number density (b) as a Ar flow rate Q_{Ar} (for microwave absorbed power P_A - 300 and 850 W).

Figure 6 shows the influence of argon flow rate on the rotational temperatures of OH radicals (a) and the electron number density (b). Naturally, increasing the gas flow caused decreasing of the temperature. As it can be seen in figure 6 the argon flow rate do not changed significantly the electron number density. Similar dependence were observed for the microwave microplasma presented in [20]. At the argon flow rate of 30 l/min and the microwave absorbed power of 300W the measured rotational temperatures of OH radicals were at the level of 800 K. In this conditions the electron number density dropped to the value of $3.3 * 10^{14}$ cm^{-3}.

Summary

The measured rotational temperatures of OH radicals ranged from 800 up to 1300 K and the electron number density ranged from $3.3 * 10^{14}$ up to $6.1 * 10^{14}$ cm^{-3}. Both of those parameters, depended on location in the plasma, and the absorbed microwave power. Moreover the temperature of heavy species strongly depended on the argon flow rate.

Preliminary tests of presented plasma shows that it have a high potential for wettability modification of various surfaces and the selected spices (e.g. juniper berries) quality improvement (microorganism colony reduction).

Moderate plasma gas temperature as well as high electron density makes presented plasma waveguide-supplied device attractive tool for different surface treatment. However more attention have to be paid on improve flow uniformity inside quartz box.

This research was supported by The National Science Centre, Poland, under the programme no. 2015/19/B/ST8/02123.

***Authors**: mgr inż. Bartosz Hrycak, mgr inż. Dariusz Czylkowski, mgr. Robert Miotk, dr hab. inż. Mariusz Jasiński, The Szewalski Institute of Fluid Flow Machinery, Polish Academy of Sciences, ul. Fiszera 14, 80-231 Gdańsk, E-mail: bartosz.hrycak@imp.gda.pl; dczylkowski@imp.gda.pl; robert.miotk@imp.gda.pl; mj@imp.gda.pl*

REFERENCES

1] Yi C.H., Lee Y.H., Yeom G.Y., The study of atmospheric pressure plasma for surface cleaning, *Surface and Coatings Technology*, 171 (2003) 237–240

[2] Kim M.C., Yang S.H., Boo J.-H., Han J.G., Surface treatment of metals using an atmospheric pressure plasma jet and their surface characteristics, *Surface and Coatings Technology* 174–175 (2003) 839–844

[3] Peelamedu R., Kumar D., Kumar S., Microwave atmospheric pressure plasma for surface treatment and reactive coating on steel surfaces, *Surface & Coatings Technology* 201 (2006) 4008–4013

[4] Hegemann D., Hossain M.M., Balazs D.J., Nanostructured plasma coatings to obtain multifunctional textile surfaces, *Progress in Organic Coatings* 58 (2007) 237–240

[5] Kondo S., Sasai Y., Kuzuya M., Development of biomaterial using durable surface wettability fabricated by plasma-assisted immobilization of hydrophilic polymer, *Thin Solid Films* 515 (2007) 4136–4140

[6] Ren C.-S., Wang K., Nie Q.-Y., Wang D.-Z., Guo S.-H., Surface modification of PE film by DBD plasma in air, Applied Surface Science 255 (2008) 3421–3425

[7] Kuzuya M., Sawa T., Mouri M., Kondo S.-I., Takai O., Plasma technique for the fabrication of a durable functional surface on organic polymers, *Surface and Coatings Technology* 169–170 (2003) 587–591

[8] Denes F.S., Manolache S., Macromolecular plasma-chemistry: an emerging field of polymer science, *Prog. Polym. Sci.* 29 (2004) 815–885

[9] Chu P.K., Chen J.Y, Wang L.P., Huang N., Plasma-surface modification of biomaterials, *Materials Science and Engineering R* 36 (2002) 143–206

[10] Morent R., De Geyter N., Verschuren J., De Clerck K., Kiekens P., Leys C., Non-thermal plasma treatment of textiles *Surface & Coatings Technology* 202 (2008) 3427–3449

[11] Tendero C., Tixier C., Tristant P., Desmaison J., Leprince P., Atmospheric pressure plasmas: A review, *Spectrochimica Acta Part B* 61 (2006) 2 – 30

[12] Czylkowski D., Hrycak B., Miotk R., Jasiński M., Compact microwave plasma device for surface treatment. 10th International Conference "New Electrical and Electronic Technologies and their Industrial Implementation" NEET 2017, Zakopane, Poland, June 27 – 30, 2017

[13] Jasiński M., Mizeraczyk J., Plasma Sheet Generated by Microwave Discharge at Atmospheric Pressure, *IEEE Trans. Plasma Sci.*, 39, 2011, 2136–2137

[14] Bruggeman P., Brandenburg R., Atmospheric pressure discharge filaments and microplasmas: physics, chemistry and diagnostics, *J. Phys. D: Appl. Phys.* 46 (2013) 464001 (28pp)

[15] Izarra Ch., UV OH spectrum used as a molecular pyrometer, *J. Phys. D: Appl. Phys.* 33 (2000) 1697-1704

[16] Raud J., Laan M., Jogi I., Rotational temperatures of N2(C,0) and OH(A,0) as gas temperature estimates in the middle pressure Ar/O2 discharge, *J. Phys. D: Appl. Phys.* 44 (2011) 345201 (5 pp)

[17] Luque J., Crosley D.R., LIFBASE: Database and Spectral Simulation Program (Version 1.5), SRI International Report MP 99-009, 1999

[18] https://www.sri.com/engage/products-solutions/lifbase (24.06.2017)

[19] Gigosos M.A., V. Cardenoso, New plasma diagnosis tables of hydrogen Stark broadening including ion dynamics, *J. Phys. B: At. Mol. Opt. Phys.* 29 (1996) 4795-4838

[20] Hrycak B., Jasiński M., Mizeraczyk J., Spectroscopic investigations of microwave microplasmas in various gases at atmospheric pressure, *European Physical Journal D* 60 (2010) 609-619

[21] Miotk R., Hrycak B., Jasinski M., Mizeraczyk J., Spectroscopic study of atmospheric pressure 915 MHz microwave plasma at high argon flow rate, *Journal of Physics Conference Series* 406 (2012) 012033 (10 pp)