



Advanced plasma treatment of polymer surfaces at atmospheric pressure for adhesion improvement

Kusano, Yukihiro; Singh, Shailendra Vikram; Drews, Joanna Maria; Norrman, Kion; Leipold, Frank; Bardenshtein, Alexander; Krebs, Niels

Published in:

Advanced plasma treatment of polymer surfaces at atmospheric pressure for adhesion improvement

Publication date:

2011

Document Version

Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

Citation (APA):

Kusano, Y., Singh, S. V., Drews, J. M., Norrman, K., Leipold, F., Bardenshtein, A., & Krebs, N. (2011). Advanced plasma treatment of polymer surfaces at atmospheric pressure for adhesion improvement. In *Advanced plasma treatment of polymer surfaces at atmospheric pressure for adhesion improvement*

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Advanced plasma treatment of polymer surfaces at atmospheric pressure for adhesion improvement

Yukihiro Kusano¹, Shailendra Vikram Singh¹, Joanna Drews¹, Kion Norrman¹, Frank Leipold¹, Alexander Bardenshtein^{1,2}, Niels Krebs²

¹ Risø National Laboratory for Sustainable Energy, Technical University of Denmark, 4000 Roskilde, Denmark

² FORCE Technology, 2605 Brøndby, Denmark

1. Introduction

Atmospheric pressure plasma processing is useful for adhesion improvement of polymer surfaces, since cleaning, roughening and addition of polar functional groups can be expected without using vacuum systems. One of the challenges in developing atmospheric pressure plasma processing is to demonstrate high productivity (high energy) and high chemical selectivity (non-equilibrium state) simultaneously [1]. Gliding arcs, which are generated between diverging electrodes in a gas flow, can fulfill the requirement [2]. Atmospheric pressure plasma processing can also be improved by ultrasonic irradiation into a plasma. Ultrasonic waves can reduce the thickness of the gas boundary layer between the plasma and the surface, and thus reactive species in the plasma can be more accessible to the surfaces.

Glass fibre reinforced polyester (GFRP) composites exhibit high strength-weight ratio and corrosion resistance and are used for a variety of applications. Adhesives are required for joining GFRPs tightly, while the adhesive joint requires careful surface preparation.

In the present work, GFRPs are treated at atmospheric pressure with (a) a gliding arc, and (b) a dielectric barrier discharge (DBD) with/without ultrasonic irradiation for adhesion improvement.

2. Experimental methods

Specimens were cut from orthophalic GFRP panels [3]. They were cleaned with acetone and methanol. The gliding arc was generated between two stainless-steel water-cooled

tubular electrodes with a diverging configuration [3,4]. Blade-shaped stainless-steel pieces were welded to the 6-mm diameter tubular electrodes, facing each other as shown in Figure 1 a). The arc discharge was ignited between these blade-shaped pieces. In order to treat a GFRP plate surface, the plate was fixed on a holder which moved forward and back at a speed of 180 mm/s. The input power to the gliding arc was fixed at 720 W. Atmospheric pressure DBD with/without ultrasonic irradiation was also used to treat GFRP plates [5-7]. The DBD was generated between two parallel plate electrodes at 100 W as shown in Figure 1 b, c). Both the gliding arc and the DBD were driven by a power supply at a frequency of approximately 40 kHz (Generator 6030. SOFTAL Electronic GmbH, Germany).

The contact angles for deionized water and glycerol on the surfaces were measured in air at room temperature for evaluation of the surface energy using a contact angle measurement system (CAM100. CreLab Instruments AB, Sweden).

X-ray photoelectron spectroscopy (XPS) was employed to analyse the elemental compositions at the surfaces. Atomic concentrations of each element were calculated by determining the relevant integral peak intensities using a linear background.

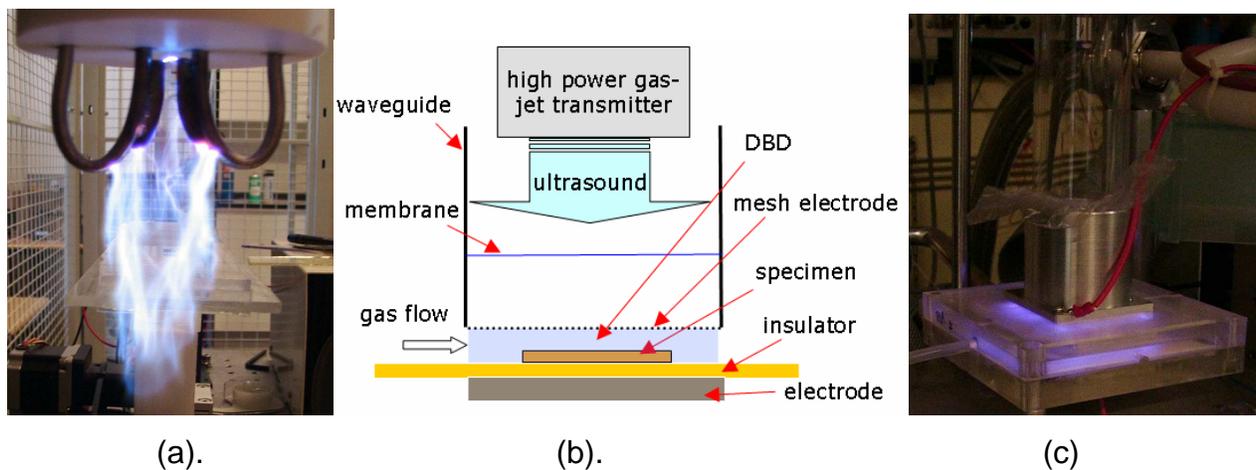


Figure 1 Setups of the gliding arc (a) and ultrasound enhanced DBD (b,c).

3. Results and discussion

The preliminary experiment of the gliding arc indicated that the temperature of the electrodes should be low enough so as to achieve stable operation at high input power. In

addition, the gas temperature at around the discharge ignition point should be high enough so that high input power with a non-equilibrium condition can be achieved [3]. After the gliding arc treatment, the wettability of the surfaces was improved, and the polar component of the surface energy and the O/C ratio at the surfaces were significantly increased. It is most likely that these changes can improve adhesion properties of the GFRP surfaces due to better interaction with adhesives. Furthermore, at an optimized condition, the gliding arc can be extended up to approximately 6 cm in the ambient air [4].

Table 1. Elemental composition (at. %) and O/C ratio at the GFRP surfaces characterized by XPS.

| | Ultrasound | Elemental composition (at. %) | | | | O/C Ratio |
|-----------------|------------|-------------------------------|------|-----|-----|-----------|
| | | C1s | O1s | N1s | Si | |
| Untreated | - | 76.5 | 22.6 | 0.6 | 0.1 | 0.30 |
| He DBD 5 s | - | 75.6 | 23.6 | 0.7 | 0.2 | 0.31 |
| | X | 69.3 | 28.0 | 0.5 | 0.5 | 0.40 |
| He DBD 30 s | - | 71.5 | 27.5 | 0.5 | 0.1 | 0.39 |
| | X | 68.3 | 30.5 | 0.2 | 0.1 | 0.45 |
| Ar DBD 30 s | - | 72.3 | 26.9 | 0.6 | 0.2 | 0.37 |
| | X | 68.3 | 30.2 | 0.3 | 0.0 | 0.44 |
| air DBD 5 s | - | 77.1 | 22.5 | 0.4 | 0.0 | 0.29 |
| | X | 70.7 | 28.4 | 0.9 | 0.0 | 0.40 |
| air DBD 30 s | - | 72.2 | 27.3 | 0.5 | 0.0 | 0.38 |
| | X | 70.8 | 28.7 | 0.5 | 0.0 | 0.41 |

It is shown that DBD treatments in He, Ar, and air without ultrasonic irradiation could oxidize the GFRP surfaces and that ultrasonic irradiation further enhanced oxidation probably due to the reduction of the thickness of the gas boundary layer. The elemental compositions of the treated and untreated GFRP surfaces are summarized in Table 1.

During the air DBD treatment without ultrasonic irradiation, occasional arcing was observed, damaging the surface significantly. After the treatment, heavily oxidized spots

were randomly distributed all over the treated surface. It was found that ultrasonic irradiation suppressed the arc ignition during plasma treatment, preventing the GFRP plate from damage. Furthermore, the GFRP surface was uniformly treated without heavily oxidized spots [5-7].

4. Conclusions

The gliding arc plasma and the ultrasound enhanced DBD were used to treat GFRP surfaces for adhesion improvement. It is indicated that the gliding arc can oxidize the GFRP surface substantially and be extended several cm so as to treat complicated 3-D structures. On the other hand, ultrasonic irradiation to DBD can enhance oxidation at the GFRP surfaces, suppress arcing and improve treatment uniformity.

Acknowledgement

This work is sponsored by the Ministry of Science, Technology and Innovation through the National Proof of Concept Funding (PoC) Scheme (grant numbers: 07-017766 and 09-076196).

References

1. Y. Kusano, Surf. Eng. **25(6)** (2009) 415.
2. A. Fridman, A. Chirokov, A. Gutsol, J. Phys. D Appl. Phys. **38** (2005) R1.
3. Y. Kusano, Int. Pat. No. 2011 073170 A1.
4. Y. Kusano, S. Teodoru, F. Leipold, T.L. Andersen, B.F. Sørensen, N. Rozlosnik, P.K. Michelsen, Surf. Coat. Technol. **202** (2008) 5579.
5. Y. Kusano, S.V. Singh, A. Bardenshtein, N. Krebs, N. Rozlosnik, J. Adhesion Sci. Technol. **24** (2010) 1831.
6. Y. Kusano, S.V. Singh, K. Norrman, F. Leipold, J. Drews, P. Morgen, A. Bardenshtein, N. Krebs, J. Adhesion **87** (2011) 720.
7. Y. Kusano, S.V. Singh, K. Norrman, J. Drews, F. Leipold, N. Rozlosnik, A. Bardenshtein, N. Krebs, Surf. Eng. In press.