Technical Report

Bonding of COP Substrates at Room Temperature Using H_2O-based Plasma Treatment

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Introduction

As the human life span and the proportion of older people are increasing, to maintain the quality of life it is very important to recognize the sign of diseases at early stage [1]. One promising means of achieving this is the "Point-of-care Testing (POCT)", which obtains, analyses and diagnoses biomarkers of different diseases from the body fluids. This means examinations can be carried out quickly at the patient's circumstances such as at home or neighbor clinic. It is considered that the POCT can improve the quality of the medical care in terms of moderating therapeutic policies and the dosage of medications by allowing timely treatment according to the results. However, existing biomarker analyses are time consuming and costly because they are carried out by specialized medical centers. Moreover, blood test requires a few ml of blood. Microfluidic chips are expected to be a new technology that enables POCT to be carried out cheaply and easily, with only a small amount of body fluids but in the meantime maintain a high sensitivity and accuracy.

While glass and silicone resins have been the mainstream of microfluidic chip materials, the cyclo-olefin polymer (COP) is gaining attention for its excellent optical properties. Particularly, its autofluorescence is almost as low as that of the glass and it can be shaped by the mold. Therefore, COP is expected to be used as fluorescence detection systems. However, COP has poor wettability and adhesiveness due to its hydrophobic surface and these properties have bad impact on the fabrication of COP microfluidic chips. Although it was reported that COP substrates can be bonded without adhesive by VUV (vacuum-ultraviolet) irradiation, the flow channel pattern may deform or the COP may increase its autofluorescence due to the heat and the pressure in the bonding process.

This report will be introducing the H_2O-based plasma (Aqua Plasma®) treatment that enables COP substrates to bond with each other at room temperature without pressing.

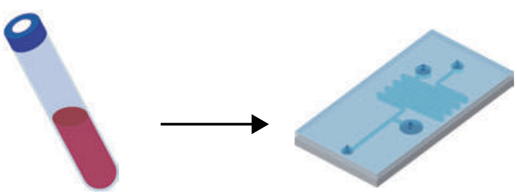

Fig.1. Biomarker assay (left) and microfluidic chip-based assay (right)

Materials and Methods

COP substrates used in this research were ZEONOR (8 mm x 30 mm, 0.5 mm thick, ZEON Corporation). The equipment used for the Aqua Plasma® treatment was Samco's AQ-2000. Experiments were carried out with different process times and fixed H_2O vapor flow rate and RF power.

After the Aqua Plasma® treatment, two COP substrates were overlaid for self-bonding at room temperature. Once the substrates bonded with each other, the bonding strength was measured by the flexural adhesion strength test (JIS K 6856 standards).

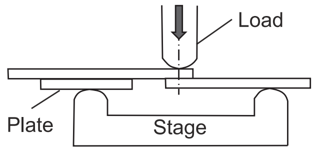

Fig.2. flexural adhesion strength test (JIS K 6856)

After the Aqua Plasma® treatment, the contact angle of pure water was measured using the contact angle meter (CA-D, Kyowa Interface Science). For comparisons, substrates treated with oxygen plasma and substrates treated with 10-minute VUV irradiation (172 nm wavelength, 10 mW/cm^2 luminance, 2 mm between the VUV source and substrate at atmosphere) were prepared.

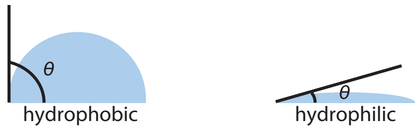

Fig.3. water contact angle

In addition, changes in the state of the chemical bonds on the surface (around 5 nm deep) of COP substrates were investigated by X-ray photoelectron spectroscopy analysis (PHI 5000 Versa Probe II, ULVAC).

Results

Bonding strengh of COP substrates at room temperature

Two COP substrates that had been treated with Aqua Plasma® successfully self-bonded with each other at room temperature by overlay. When 1 N/cm^2 of pressure was added to the substrate, the substrate on the top broke before it was flaked off the other substrate. Whereas substrates treated with VUV irradiation and oxygen plasma did not self-bond with each other without heating.

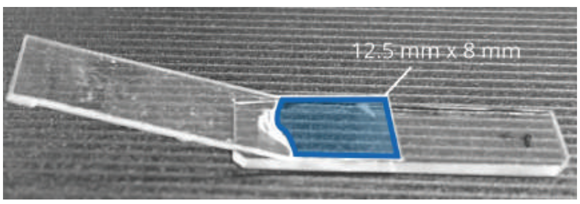

Fig.4. A pair of bonded COP substrates which were destroyed during the flexural bonding strength test

COP surface wettability

Fig.5 shows water contact angle after Aqua Plasma® treatment, UVV irradiation or oxygen plasma treatment.

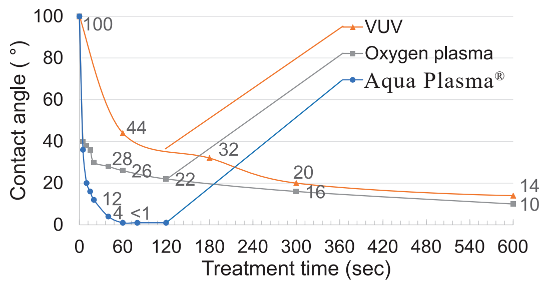

Fig.5. wafer contact angle after surface treatment

In order to maximize the liquid fluidity, the water contact angle of the flow channel should be as low as possible. The contact angle of the COP substrates treated with VUV irradiation and oxygen plasma saturated at around 10°. On the other hand, it is called “super-hydrophilic” if the contact angle is less than 5°. Low contact angle of 1° was achieved by processing the COP substrate for 60 seconds with Aqua Plasma®.

Status of the checmical bonds on the COP surfaces

Functional groups were detected from the surface of COP substrates that were treated with Aqua Plasma® or oxygen plasma. However, the portion of the -COOH on the substrate treated with Aqua Plasma® was 3 times of that of the oxygen plasma. Therefore, we surmised that -COOH plays an important role in achieving bonding at room temperature and super-hydrophilicity.

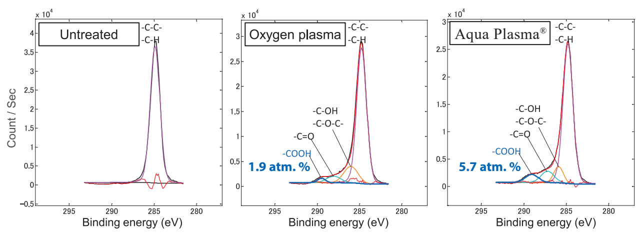

Fig.6. C1s spectra of COP before and after surface treatment

Summary

This report has illustrated the effects of the Aqua Plasma® treatment. By treating the surface of COP substrates with Aqua Plasma®, two substrates could bond with each other at room temperature and the bonding strength was enough for microfluidics fabrication. Moreover, after the Aqua Plasma® treatment, the surface became super-hydrophilic with a high portion of -COOH. It is already known that OH, H and O radicals are generated by Aqua Plasma® so we surmise that these radicals react with the surface of the COP substrate to form the -COOH functional group, which contributes to the bonding at room temperature and the super-hydrophilicity.

Reference

[1] R. Ishihara, K. Hasegawa, K. Hosokawa, and M. Maeda, “Detection of Proteins and Nucleic Acids with Laminar Flow-Assisted Dendritic Amplification on Power-Free Microfluidic Chip” (in Japanese) ***Bunseki Kagaku***, vol. 64 no. 5, pp. 319–328, May 2015.

[2] H. Terai, R. Funahashi, and M. Kakuta, “Room-temperature Bonding of COP Substrates Using Aqua Plasma (Water Vapor Plasma)” (in Japanese) ***CHEMINAS Abstracts***, vol. 35th, p.47, 2017.

※ Aqua Plasma® Trademark No.5899818
※※ Patent pending in five countries including Japan