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Direct bonding of biaxially oriented polyethylene terephthalate films with plasma surface modification using various gases

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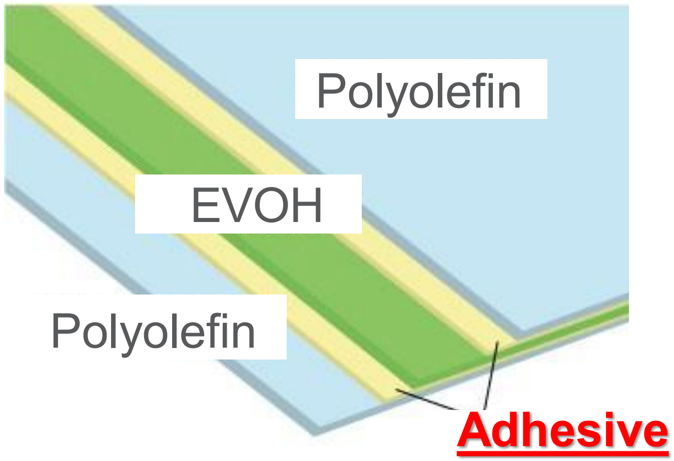
1. Background

Examples of laminating plastic films used in industry

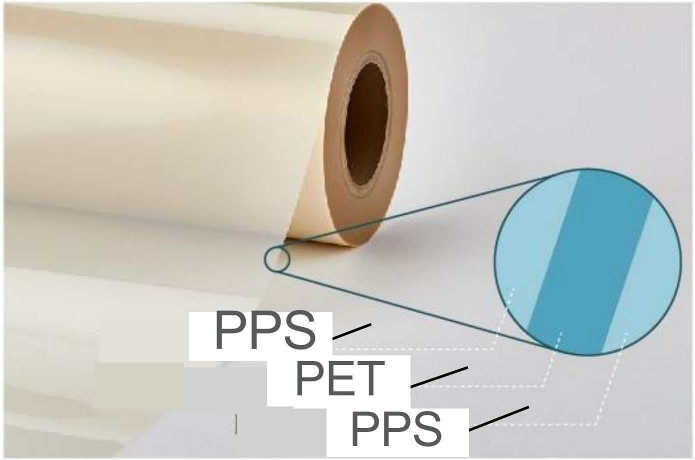
Multilayer Barrier Film



https://www.ube.co.jp/ubefilm/products/order/barrier_film.html

- To improve gas barrier properties
- To boost strength

Heat-resistant Insulating Film



https://www.films.toray/products/tlt/

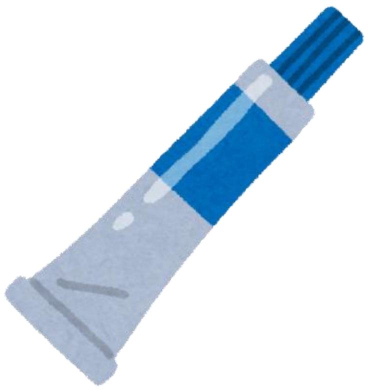
- To enhance electrical insulation
- To optimize heat resistance

Plastic film manufacturers are laminating plastic films with different properties using adhesives to obtain the required characteristics.

1. Background

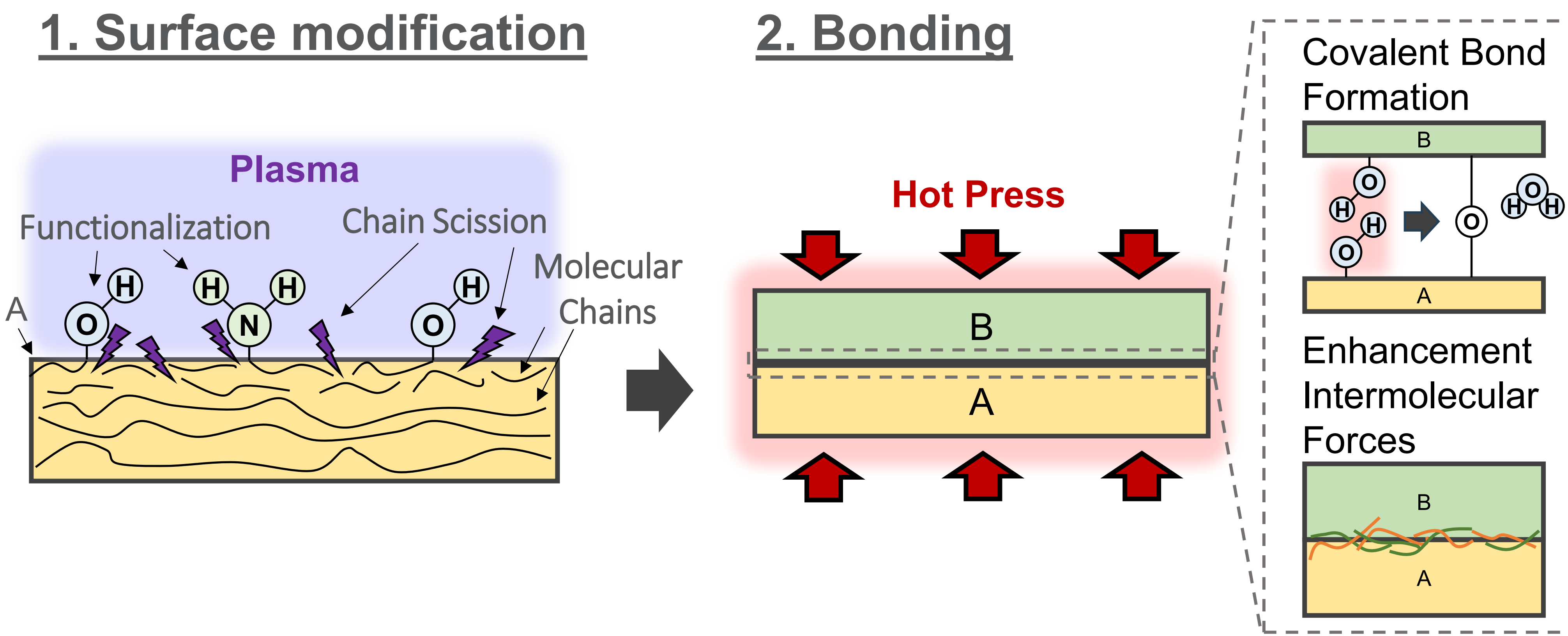
Disadvantages of using adhesives

- Peeling due to adhesive deterioration
- Elution of adhesive components
- Increase in thickness due to the adhesive layer



To solve the above issues, we focused on adhesive-free bonding technology (direct bonding).

2. Introduction to Direct Bonding



In the case of direct bonding technology using plasma, it has been reported that direct bonding can be achieved by heat pressing after plasma surface modification.

3. Research Objective

In previous research

It has been reported that PET films can be directly bonding by vacuum plasma surface modification using **O_2** gas.

Tamio Endo et al., "Composite engineering – direct bonding of plastic PET films by plasma irradiation", Procedia Engineering, 171 (2017) 88-103

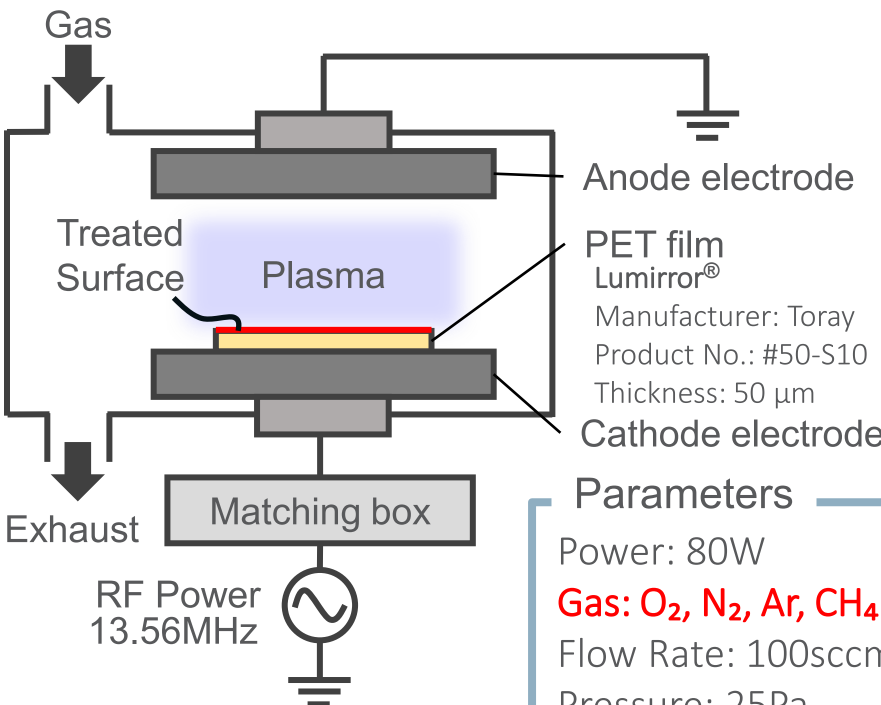
However, the effects of gas species used in vacuum plasma surface modification have not been investigated.

In this study

We investigated the direct bonding of PET films modified by vacuum plasma surface modification using various gas species (**N_2, Ar, CH_4**).

4. Conditions for Direct Bonding

① Vacuum Plasma Treatment



Parameters

Power: 80W

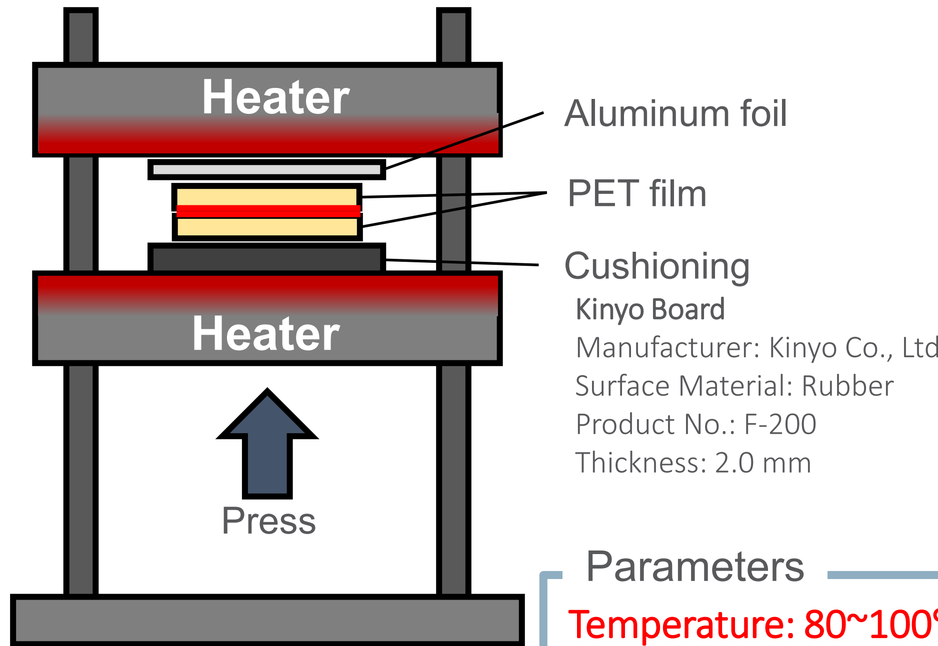
Gas: O_2, N_2, Ar, CH_4

Flow Rate: 100sccm

Pressure: 25Pa

Treatment Time: 30s

② Hot Press



Parameters

Temperature: 80~100℃

Pressure: 2MPa

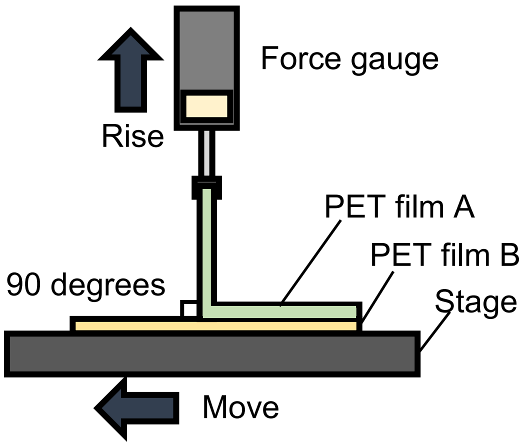
Press Time: 1min

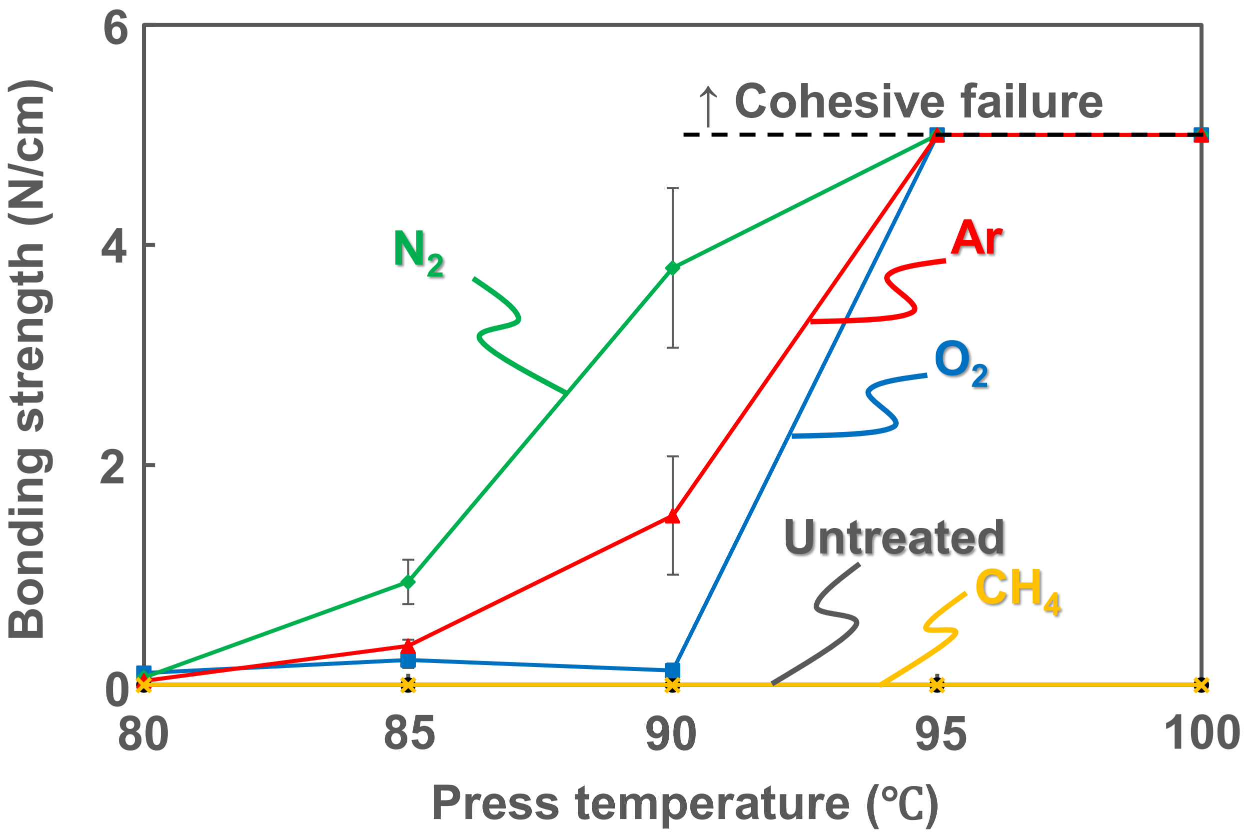
In this experiment, the gas species for vacuum plasma surface modification and hot press temperature were varied.

5. Results of Adhesive Strength

<u>Equipment</u>
Manufacturer: IMADA
Model: ZTS-5N, MX2-500N

<u>Measurement Conditions</u>
Peel speed: 50mm/min
Substrate width: 1cm
n=3



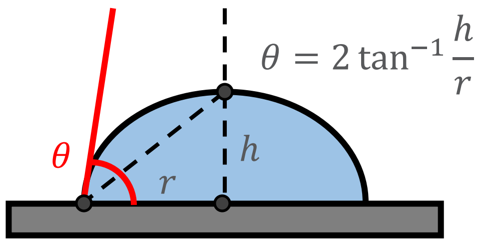


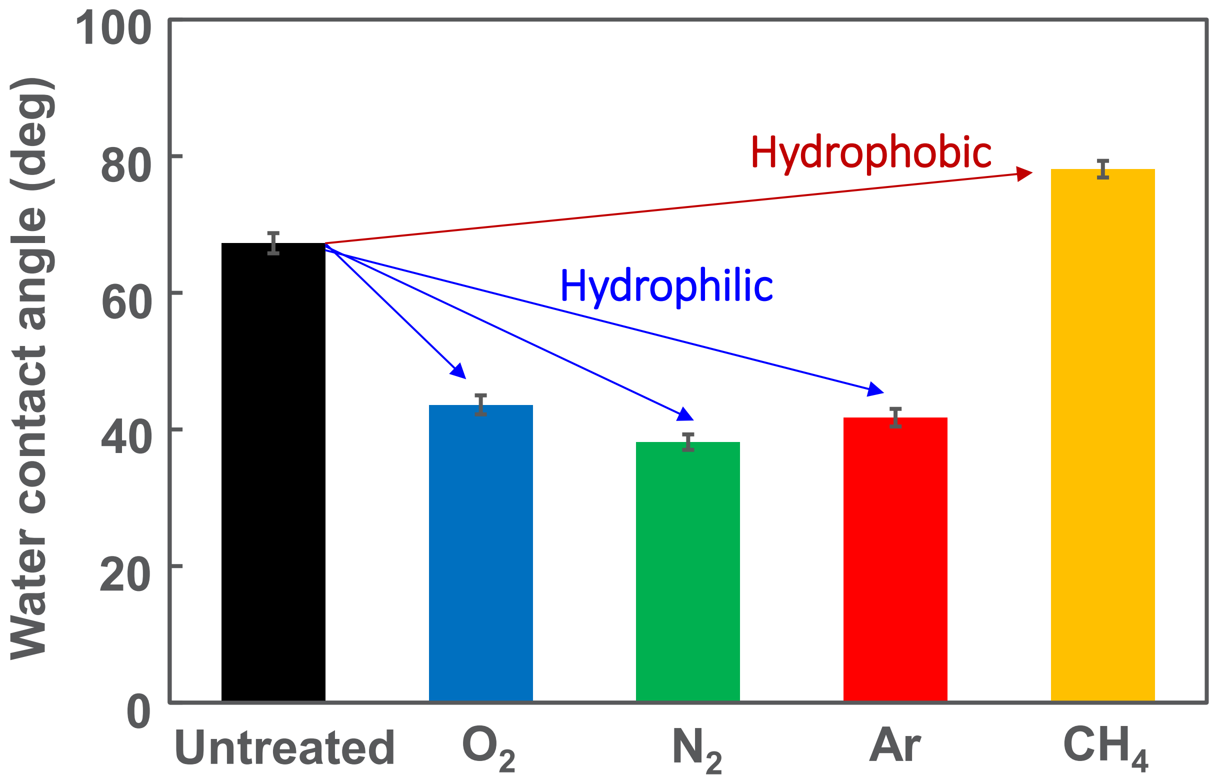
This bond strength increases with the heat press temperature, and we confirmed that the gas species in vacuum plasma surface treatment are effective in the order of N_2, Ar, and O_2.

6. Results of Wettability of PET Film before Bonding

Equipment
Manufacturer: Kyowa Interface Science Co., Ltd.
Model: CA-X

Measurement Conditions
Method: θ/2 method
Droplet Volume: 0.4µL
Liquid: Pure water
n = 5





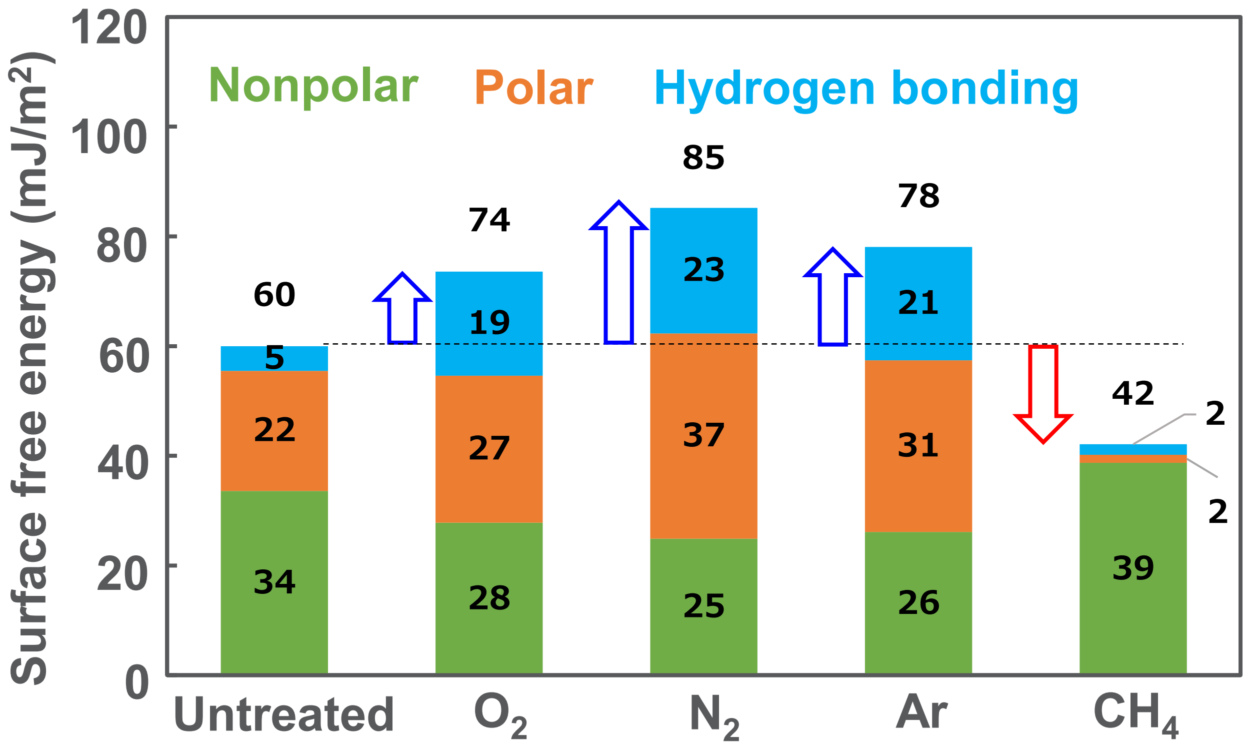
For O_2, N_2, and Ar gases, it was confirmed that the PET film surface was hydrophilic.
On the other hand, for CH_4 gas, it was found to be hydrophobic.

7. Calculation of Surface Free Energy of PET Film before Bonding

Extended Fokes equation (Kitazaki-Hatake model)

$$(1+\cos\theta)\cdot\gamma_L = 2\left(\sqrt{\gamma_S^d\cdot\gamma_L^d}+\sqrt{\gamma_S^p\cdot\gamma_L^p}+\sqrt{\gamma_S^h\cdot\gamma_L^h}\right)$$

θ : Measured contact angle
γ_L : Total surface tension value of the liquid
γ_L^d : Dispersive component of liquid
γ_L^p : Polar component of liquid
γ_L^h : Hydrogen bonding component of liquid
γ_S^d : Dispersive component of sample
γ_S^p : Polar component of sample
γ_S^h : Hydrogen bonding component of sample



It was found that the surface free energy increased in samples treated with O_2, N_2, and Ar gases, while it decreased in samples treated with CH_4 gas.

→This increase in surface free energy may have contributed to the bonding strength.

8. Results of Surface Roughness of PET Film before Bonding (AFM)

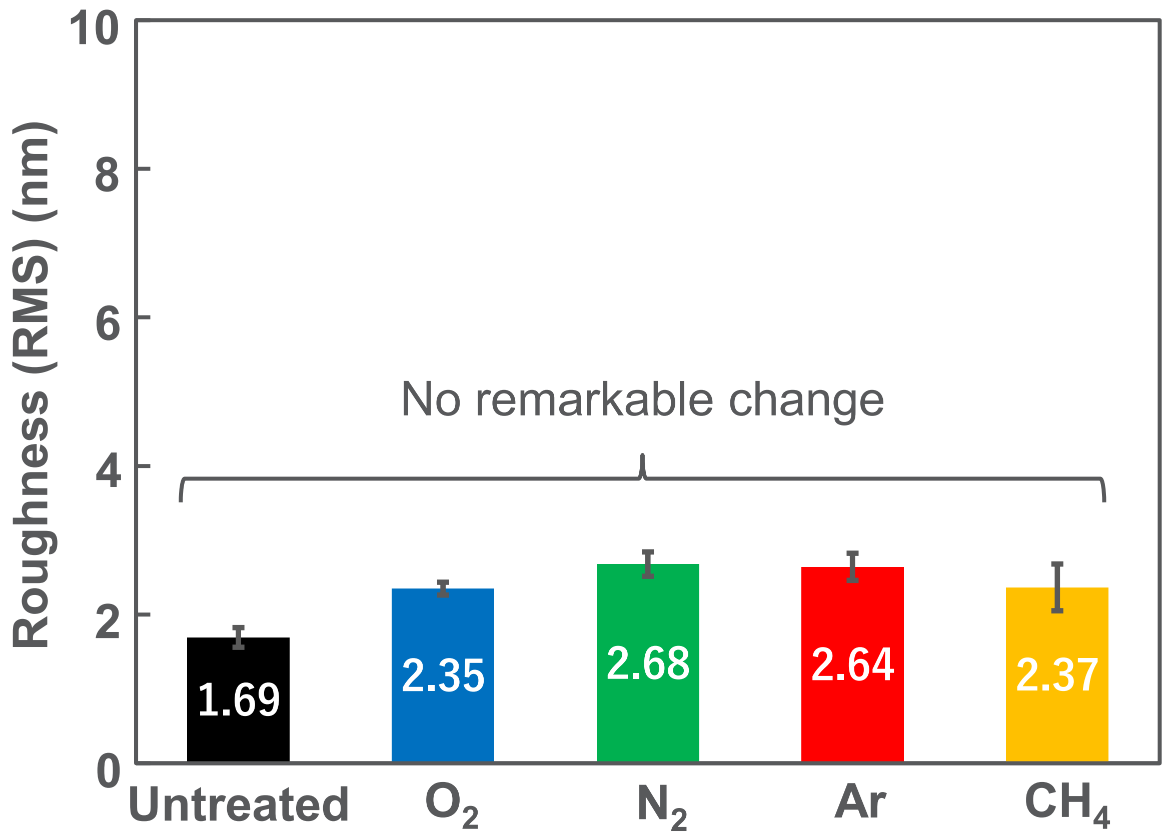
Equipment
Manufacturer: JEOL
Model: JSPM-5200

Measurement Conditions
Scan size: 1.50µm × 1.50µm
Filter: 0.5Hz
Loop gain: 16
n=3

Root mean square (RMS) surface roughness

$$\mathrm{RMS} = \sqrt{\frac{1}{N}\sum_{i=1}^{N} z_i^2}$$

z_i : Deviation in height values at individual points
N : Number of measurement points



Generally, surface free energy is influenced by physical and chemical properties, but the surface roughness of the samples was similar.

9. Results of Surface Composition of PET Film before Bonding (XPS)

Equipment

Manufacturer: Shimadzu Corporation

Model: ESCA-3400

Measurement Conditions

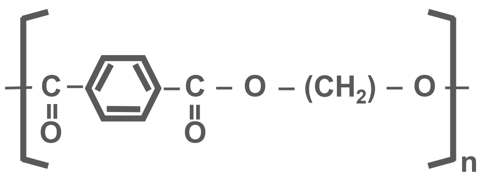
X-ray source: Mg Kα

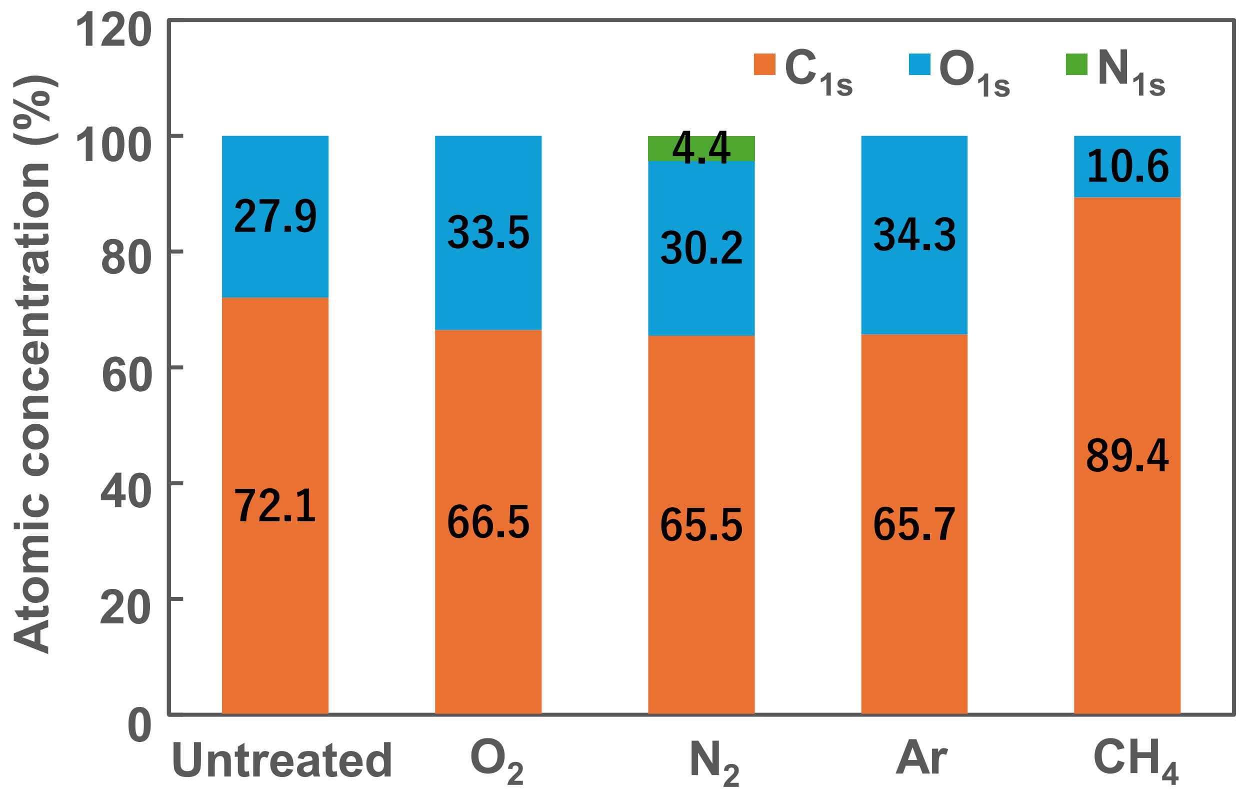
Emission current: 10 [mA]

Accelerating voltage: 10 [kV]

Energy resolution: 0.1 [eV]

Molecular structure of PET





Next, we analyzed the surface composition and found that samples treated with O_2, N_2, and Ar gases had higher oxygen content than untreated samples. In addition, nitrogen was detected in samples treated with N_2 gas.

9. Results of Surface Composition of PET Film before Bonding (XPS)

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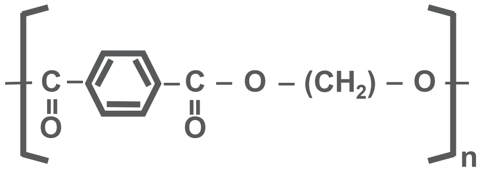

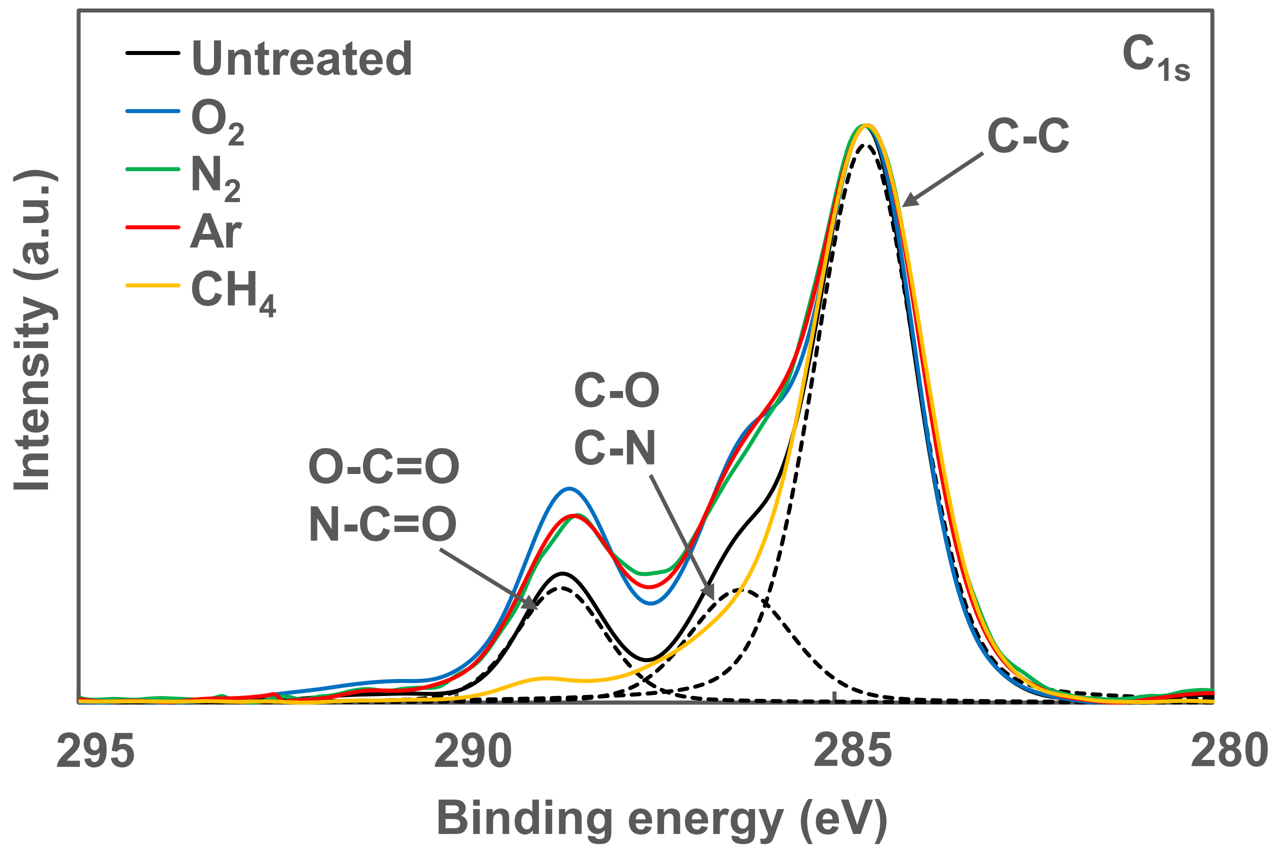

Furthermore, when the C spectrum was separated into waveforms, it was found that the polar groups C–O, C–N, and O–C=O bonds increased in samples treated with O_2, N_2, and Ar gases.

10. Conclusions

In this study

We investigated the direct bonding of PET films modified by vacuum plasma surface treatment using various gas species (N_2, Ar, CH_4).

The adhesive strength increased in the order of O_2, Ar, and N_2 for the vacuum plasma surface treatment gas types.

This difference in bonding strength may be influenced by an increase in surface free energy caused by the formation of polar groups.

Thank you!

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Sponsors / Partners:

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In this study, We used the Atomic Force Microscope (JSPM-5200) and X-ray Photoelectron Spectroscopy (ESCA-3400) at Tokyo Denki University Analysis Center