

# Method for releasing the carbon foil from the substrate

September 27, 2022

30<sup>th</sup> World Conference of the INTDS  
Villigen, Switzerland

○ Hiroo Hasebe, Hiroki Okuno, Hiroshi Imao,  
Nobuhisa Fukunishi, Masayuki Kase, and Osamu Kamigaito  
Nishina Center for Accelerator-Based Science  
RIKEN

# Table of Contents

- 1. Introduction**
- 2. Releasing agent in the arc discharge method**
- 3. Releasing agent in magnetron sputtering method**
- 4. Contact angle measurements of for various substrates**
- 5. For better releasing agents**
- 6. Summary**

# Introduction

- The carbon foils (C-foils ) ( $10 \mu\text{g}/\text{cm}^2 \sim 10 \text{mg}/\text{cm}^2$ : 0.05 to 5  $\mu\text{m}^{\text{t}}$ ) are manufactured by the vapor deposition on the glass substrate or Si wafer.
- Use releasing agents between the substrate and the foil to remove the carbon layer.
- The releasing agent is hydrophilic because the foil easily floats on water and is attached to a holder.
- However, due to its hydrophilicity, the humidity affects the foil quality.
- Chloride, soap (surfactant), and betaine saccharose are well-known releasing agents.

# Beginning of the development



Receiving inspection

Releasing agent is very important.  
Tested releasing agents:

1. Chloride
2. Soap (surfactant)
3. Sugars: Betaine saccharose  
The method from GSI.
- 4....I tried granulated sugar, the deposition chamber was wrapped in a sweet scent.

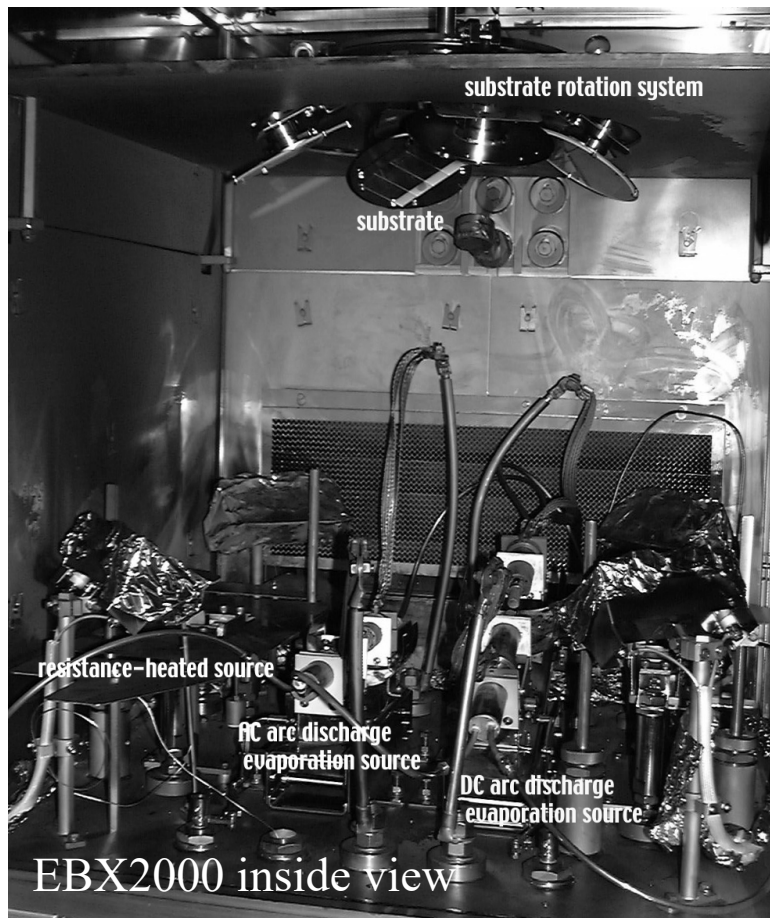
Installation of EBX2000 December 1999

Manufactured by ULVAC

Adviser: Prof. Sugai -san

Fabrication of RIKEN-original thin C-foil started.

# Releasing agent in the arc discharge method



Thickness ( $\mu\text{g}/\text{cm}^2$ )	Method	Releasing agent
10-20	AC arc 450A	$\text{NiCl}_2$
40-60	AC arc 300A	$\text{NiCl}_2$ or $\text{LaCl}_3$
80	AC+DC+AC 300A	$\text{LaCl}_3$

- In the arc discharge method, a chloride-based releasing agent using a resistance-heated source worked very well.
- $\text{NiCl}_2$  and  $\text{LaCl}_3$  (powder) are the best, depending on the C-foil thickness.
- The thickness of the releasing agent is equivalent to  $10 \mu\text{g}/\text{cm}^2$  of carbon.  
(measured by crystal rate thickness monitor)
- Floating foils on water are attached to the holders.



# Deposition of C-foil thicker than 0.1 mg/cm<sup>2</sup>

Magnetron sputtering deposition

(SH-350 by ULVAC : August, 2009)

evaporation chamber  $\phi 500 \times 300 \text{ mm}^h$

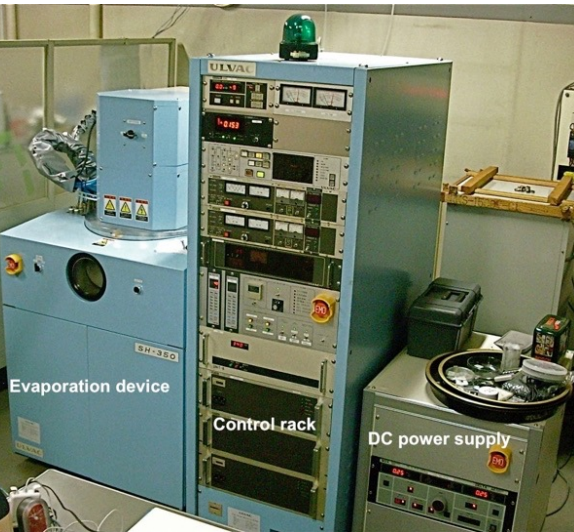
35 - 85 mm

6-inch  
Graphite target

Plasma  
Ar gas + DC or RF

Substrate  
5-inch silicon wafer  
or glass

Rotates and heated 250 °C max



# Glass substrate with C-foil



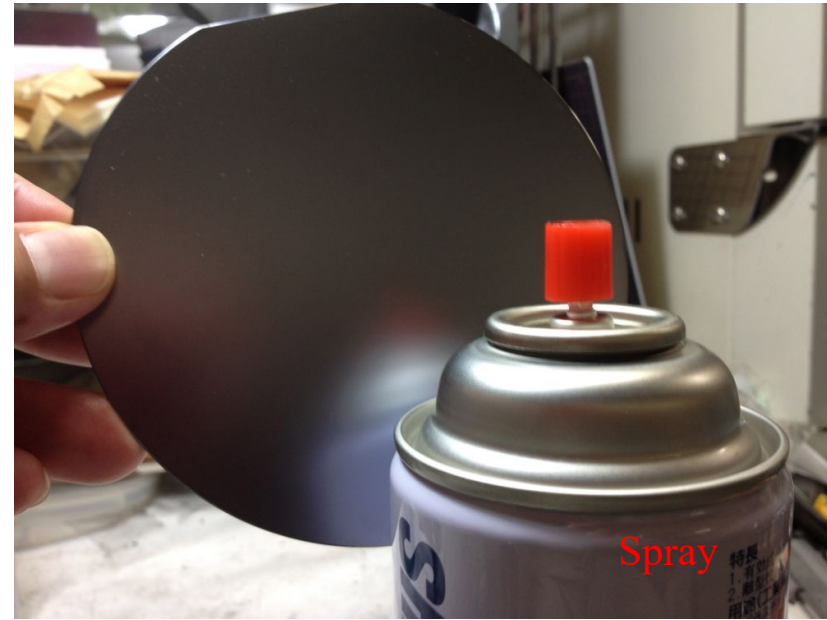
$0.3 \text{ mg/cm}^2$   
mirror surface

Thickness  
distribution  
 $\pm 1\%$

The mirror surface does not change  
(even C-foil is separated from the substrate)



# Releasing agent used and how to handle



DAIFREE by Daikin for 0.1 ~ 0.3mg/cm<sup>2</sup>  
RELEASE by Shin-Etsu Silicone for >0.3mg/cm<sup>2</sup>  
DAIFREE(GA-6310) has a changed model number

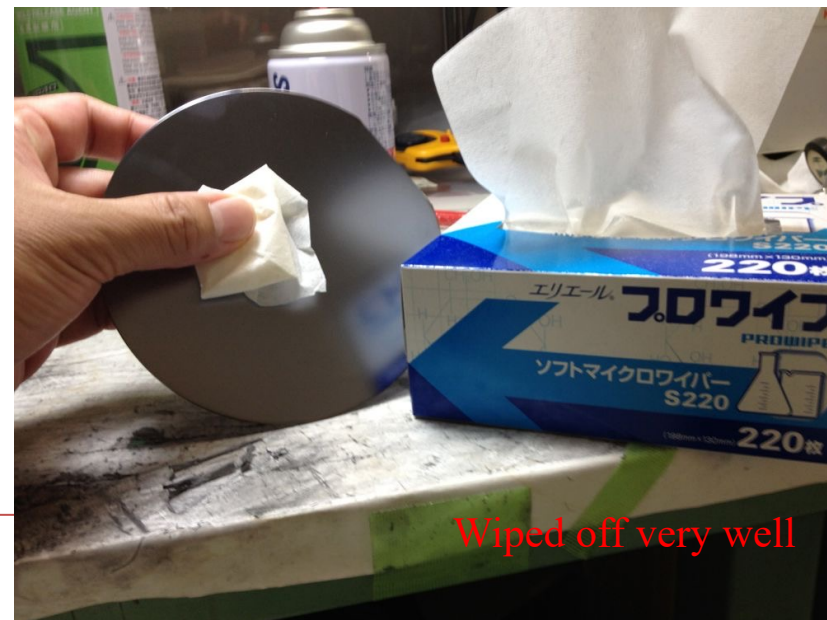
How to handle:

Spray on the substrate

wipe off with paper thoroughly!

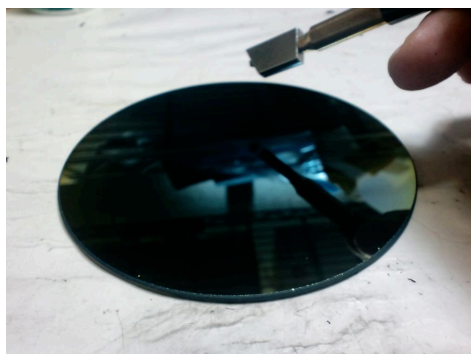
<http://www.shinetsusilicone-global.com/products/type/mold/index.shtml>

[https://www.daikinchemicals.com/solutions/products/mold-release-agents.html?\\_ga=2.182349347.544690582.1654064576-497526060.1653898399](https://www.daikinchemicals.com/solutions/products/mold-release-agents.html?_ga=2.182349347.544690582.1654064576-497526060.1653898399)

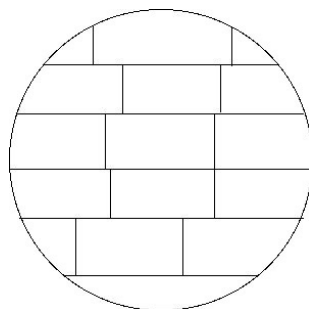
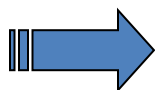




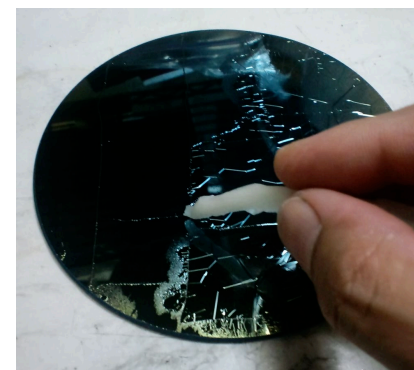
# Procedures to attach C-foil on a fixed holder



5-inch glass substrate



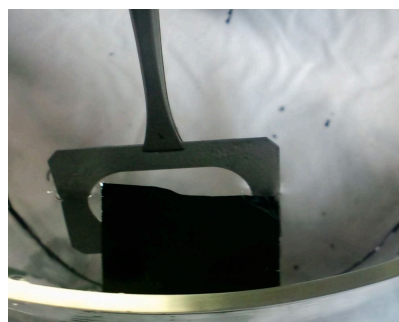
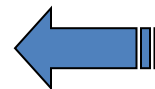
cut



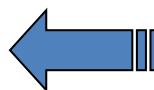
water  
penetrates



floated

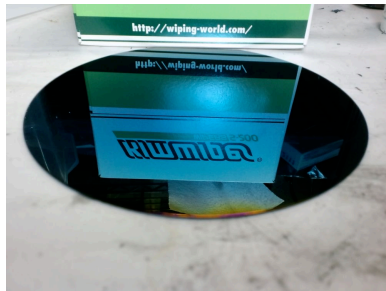


scoop



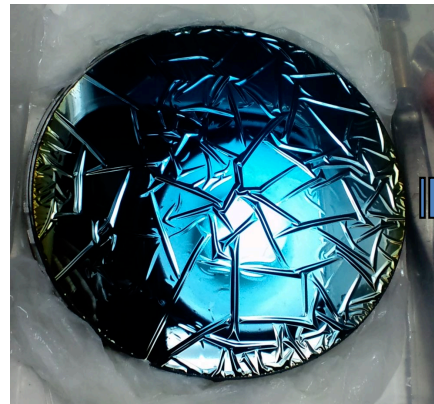
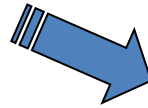
Fixed holder

# Procedures to stick C-foil on a Rotating-cylinder holder

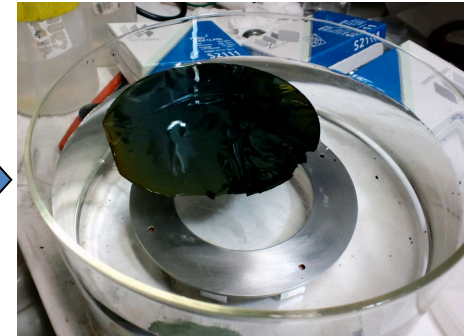


5-inch

Si wafer substrate



water penetrates



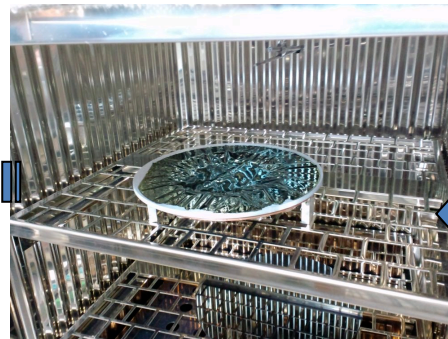
floats



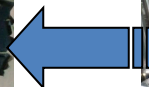
scoop



Rotating-cylinder holder



Oven 25 °C



# Polymer coating

## Poly-monochloro-para-xylylene (Parylene-C)

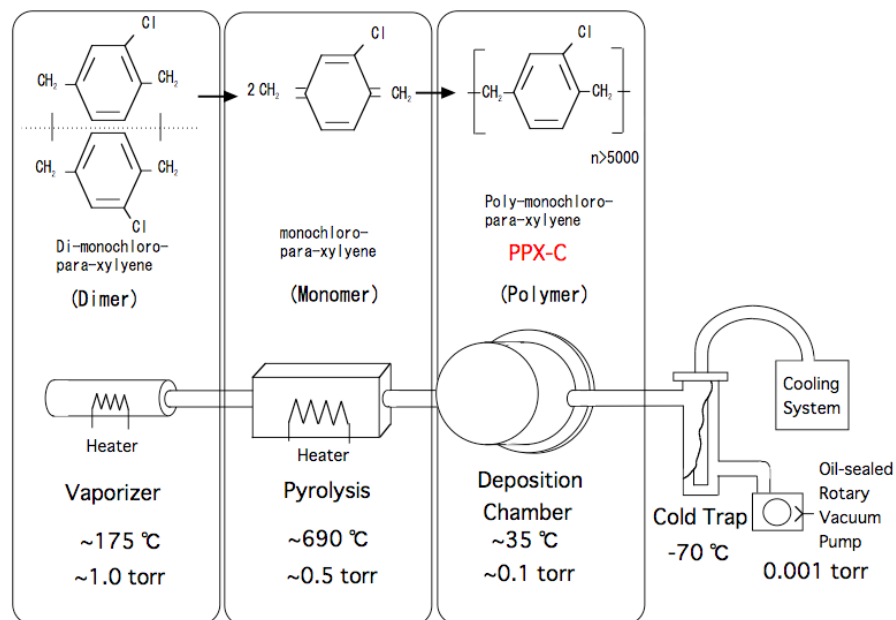


Introduced in 2005

LABCOTER (PDS 2010 )

Large-area C-foil fabrication has been required.

Successful large-area C-foil !!!



Reference : H.Hasebe et al.,  
Nucl. Instr. and Meth. A **590**, 13 (2008)

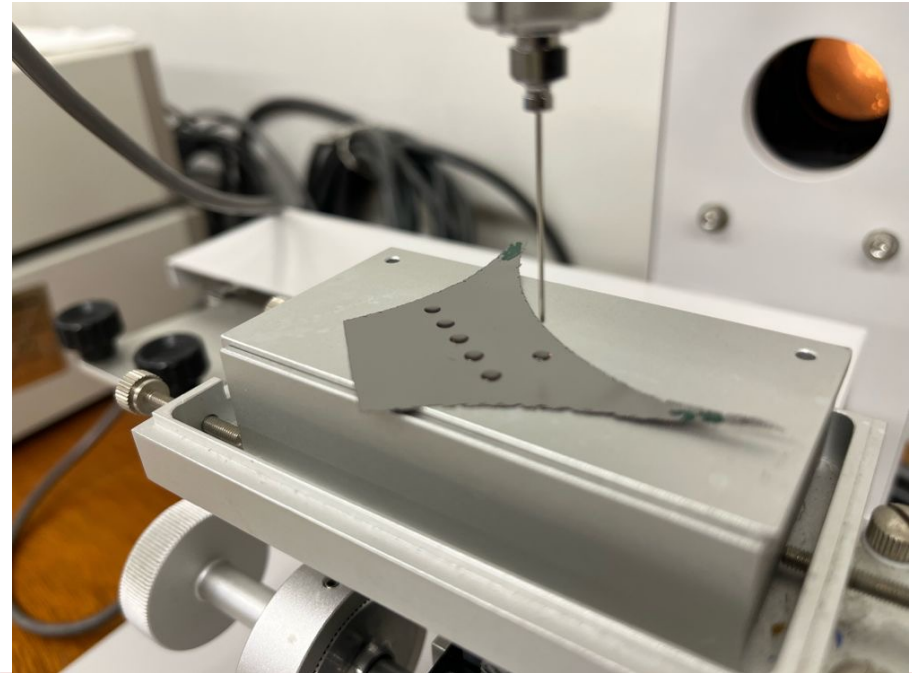
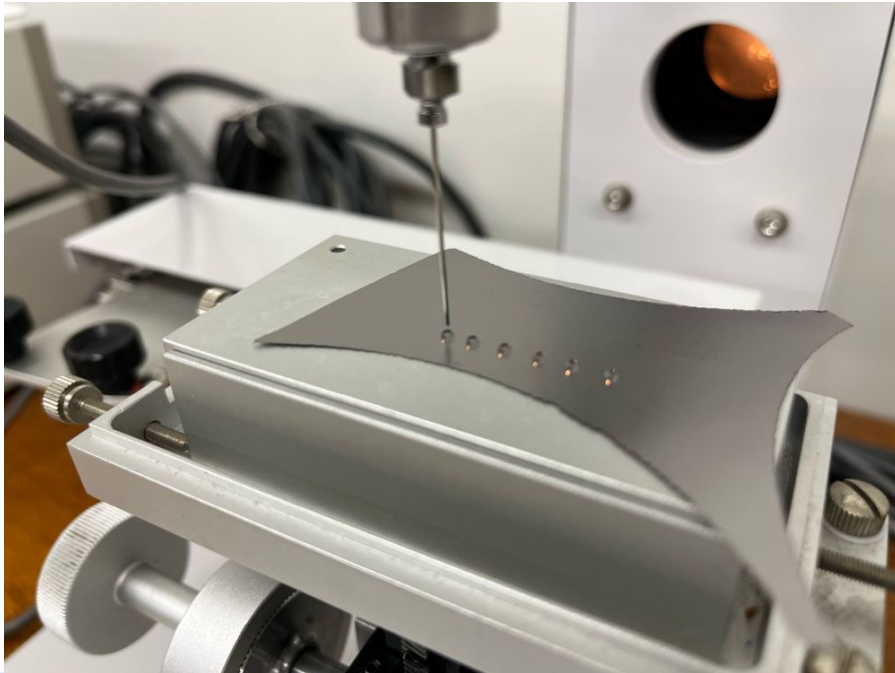


# Contact angle measurements for various substrate

## KYOWA interFace Measurement and Analysis System FAMAS

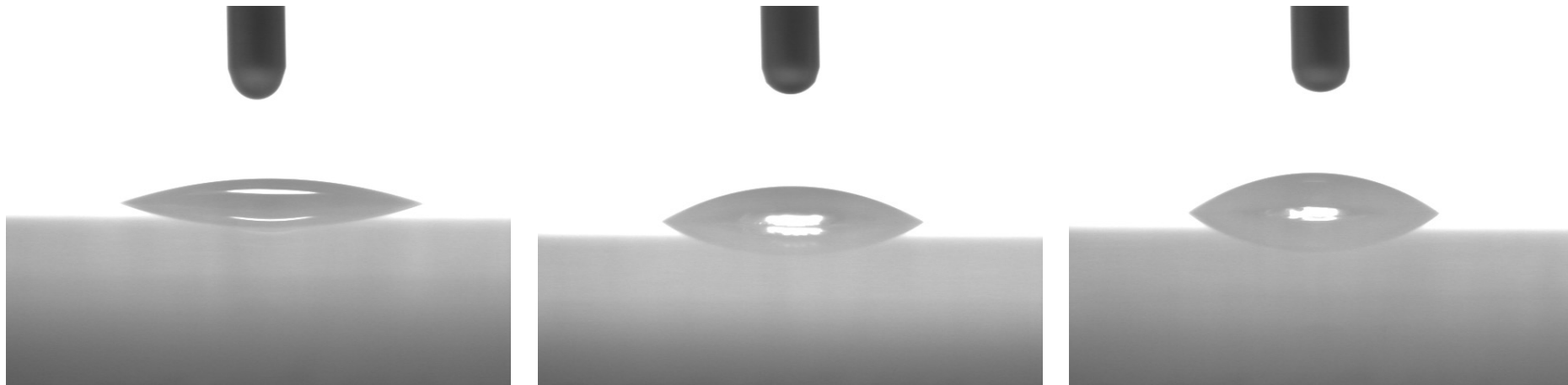


Contact angle: wetness  
Sessile drop method  
Liquid: pure water  
Liquid volume: 2  $\mu\text{L}$   
Waiting time: 5 seconds





# Contact angle of slide glass

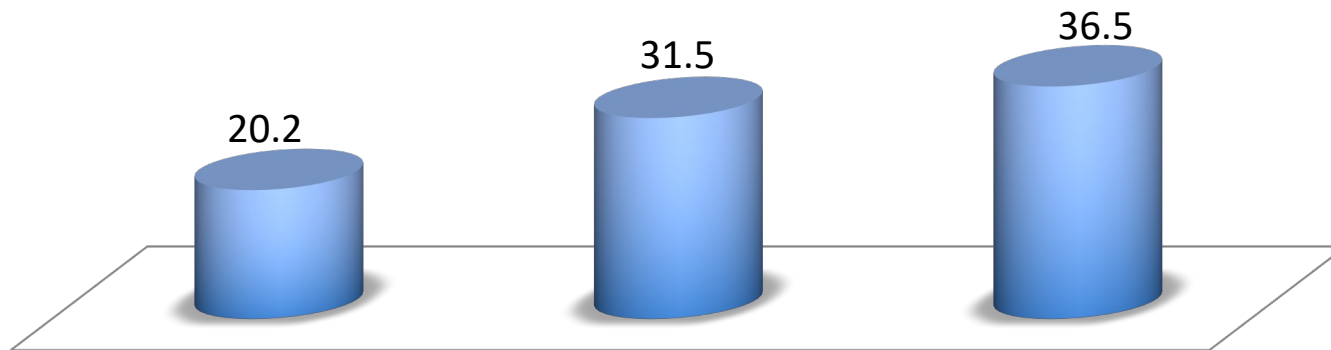


Untreated  
C.A. 20.2 deg

$\text{NiCl}_2$   
31.5

$\text{LaCl}_3$   
36.5

## Contact angles of slide glass

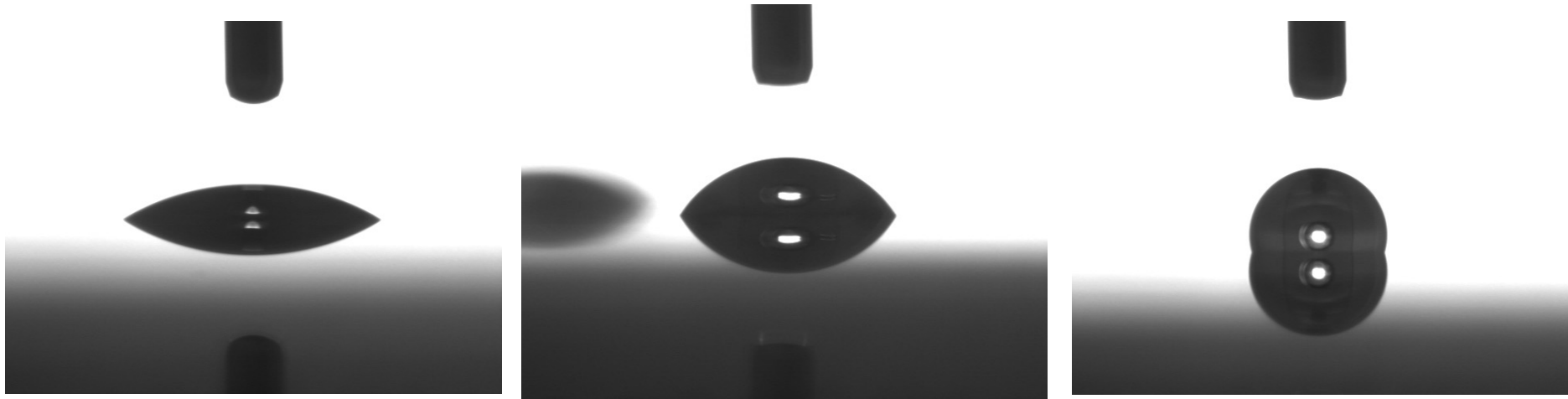


Untreated

$\text{NiCl}_2$

$\text{LaCl}_3$

# Contact angle of Si wafer

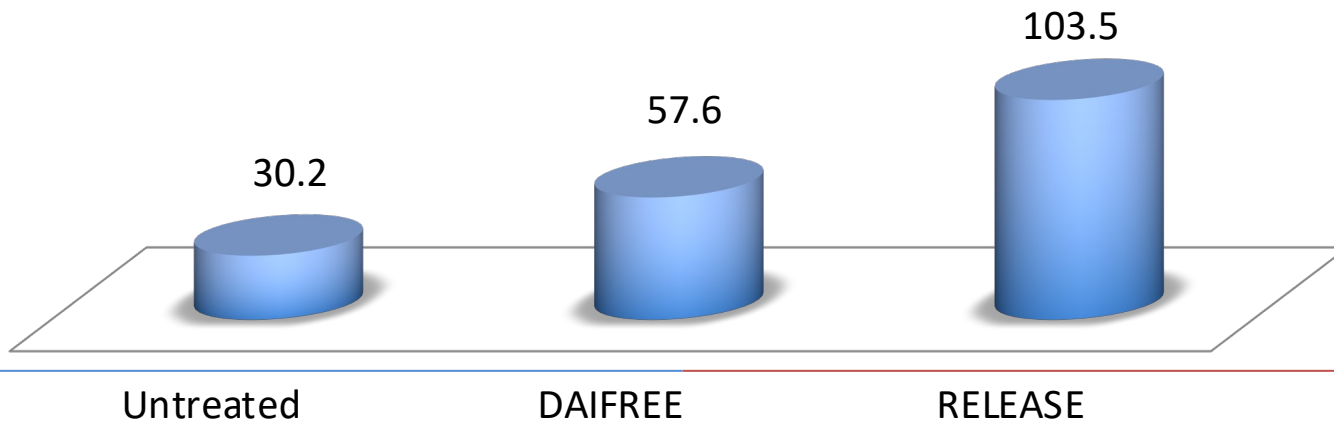


Untreated  
C.A.30.2 deg

DAIFREE Fluorine-based  
57.6

RELEASE Silicon-based  
103.5

## Contact angles of Si wafer



# Summary of contact angles by thickness

Thickness ( $\mu\text{g}/\text{cm}^2$ )	Method	Releasing agent	Substrate	Contact Angle (deg)
10-40	AC arc	$\text{NiCl}_2$	Slide glass	31.5
60-80	AC+DC+AC	$\text{LaCl}_3$	Slide glass	36.5
100-300	Magnetron sputtering	DAIFREE Fluorine-based	5-inch Si wafer	57.6
300-10000	Magnetron sputtering	RELEASE Silicon-based	5-inch Si wafer	103.5

As the C-foil thickness increases, the contact angle increases  
Negative relationship between the C-foil thickness and the **wettability** of the substrate.

# Other releasing agents

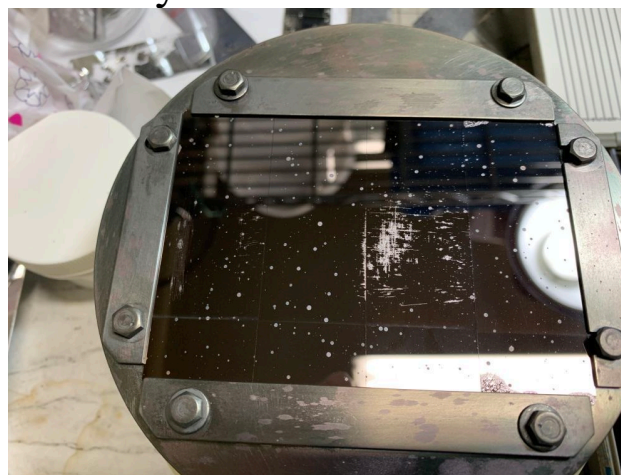
## Ionic Liquid (IL): potential candidate

- hardly evaporates at room temperature and even in vacuum.
- has Electrical conductivity.

→Development has been very active in recent years.

We tested whether the water-soluble IL can be used as a releasing agent.

Most IL is toxic and should be handled carefully.



1. Put IL in a deep Ta boat and cover it with a perforated Ta lid
2. Evaporate IL carefully so as not to boil
3. Evaporate C-foil
4. Take the C-foil out of the device and float it on water

The C-foil condition is not good because the IL amount was not optimized. IL amount was not able to be measured with the thickness monitor.



# Summary

1. Chloride is the most suitable releasing agent for C-foil with thickness 10-80  $\mu\text{g}/\text{cm}^2$ . The material is selected according to the thickness.
2. We found that fluorine-based and silicon-based releasing agents are good for C-foil thicker than 0.1  $\text{mg}/\text{cm}^2$ .
3. The damages caused by humidity were greatly reduced when applying the method above.
4. There is a relationship between the C-foil thicknesses and the contact angles of the substrate for each releasing agent.
5. IL has a possibility to be the new candidate as a good releasing agent.

[hasebe\[at\]riken.jp](mailto:hasebe@riken.jp)