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UNIVERSITÉ DU QUÉBEC À CHICOUTIMI



Hydrophobic and anti-ice properties of homogeneous and heterogeneous nanoparticle coatings on Al 6061 substrates

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Outline

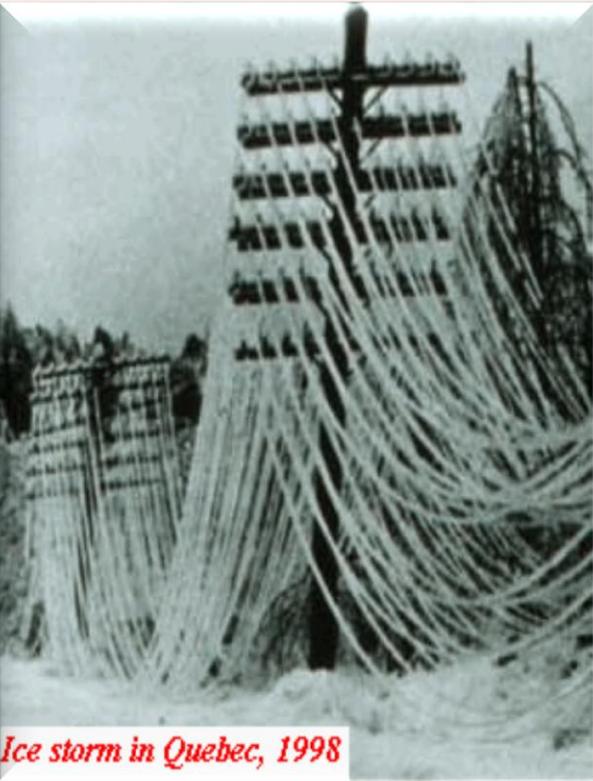
- *Introduction*
- *Background*
- *Objectives*
- *Methodology*
- *Results and Discussion*
- *Summary*

Introduction

- o Icing on structures in cold climate countries



Introduction



Introduction

A. De-icing methods

- ✓ *Mechanical de-icing*
- ✓ *Chemical de-icing*
- ✓ *Thermal de-icing*

Advantage

- *Widely used and applicable compare to anti-icing ones*

Disadvantages

- *Frequency of application,*
- *Cost issues*
- *Significant negative environmental impacts (toxicity)*



Main disadvantage

- *Using after ice build-up on structures*

Introduction

B. Anti-icing methods

✓ *Anti-icing or ice-phobic coatings*

Main advantage

➤ *Prevention ice accumulation on a surface in advance*

Advantages

- *Environmentally friendly compared to de-icing methods,*
- *Long service-life (durability),*
- *Significantly reduction of ice adhesion strength,*
- *Good cost effectiveness*



(a) Uncoated Aluminum



(b) Coated Aluminum

Disadvantage

➤ *Limited used and applicable compare to de-icing methods*

Background

❖ *Anti-icing or ice-phobic coatings*

General approach and surface treatments for icephobicity:

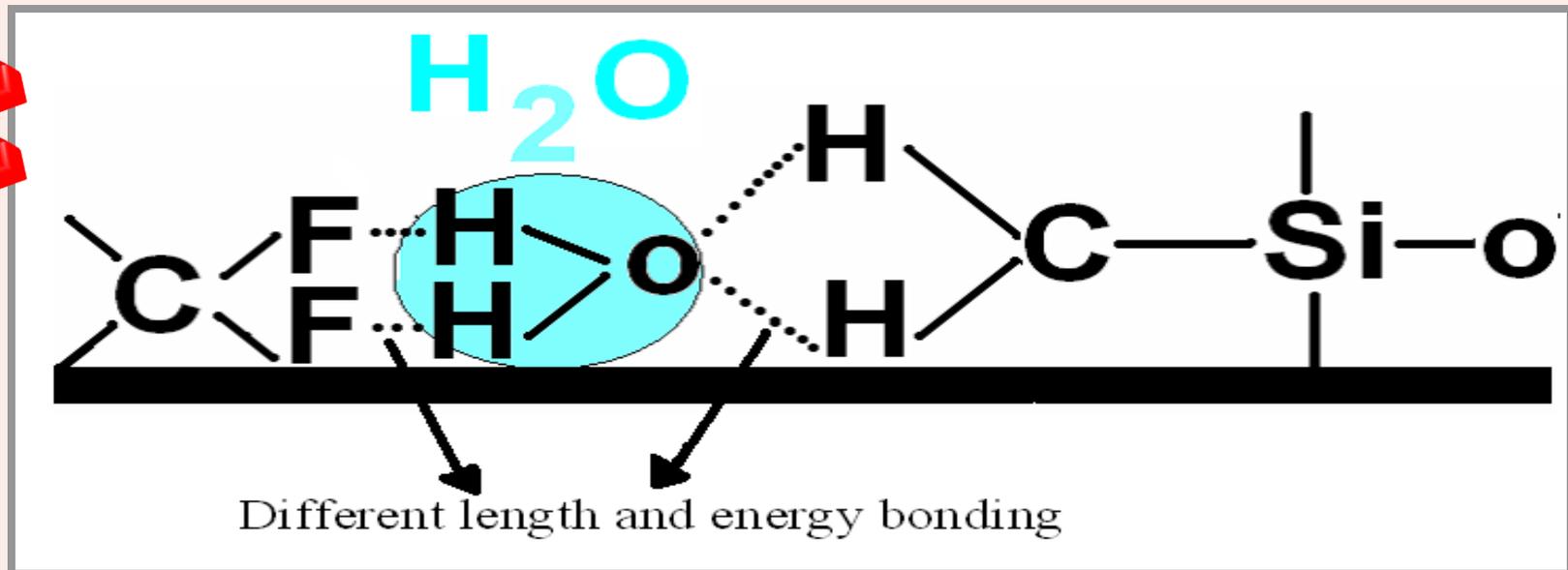
- (i) Self-assembled monolayers with $-CH_3$ or $-CF_3$ groups oriented outward to the ice surface,*
- (ii) Introducing micro-/nano scale roughness on substrate surface to increase air pockets density follow by applying low surface energy materials,*
- (iii) Coatings with heterogeneous chemical composition of at least two hydrophobic components to disturb the structure of the liquid-like layer.*

Background

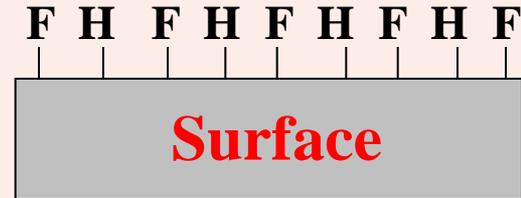
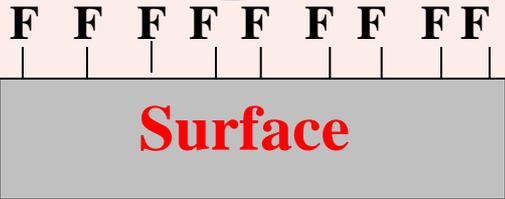
➤ *Heterogeneous coating*

- 1) *Polyperfluoroalkyl(meth)acrylate combined with hydrophobic silicon dioxide (A),*
- 2) *Organopolysiloxane modified with a lithium compound (B).*

..... compared to PTFE (homogeneous coating)



Heterogeneous *coating*



Homogeneous coating

Heterogeneous coating

Background

➤ Theoretical investigation of heterogeneity effect

Homogeneous coating

H. Murase et al.

Interaction Forms

Bond length (nm) (OH, FH)	0.252	0.329	0.187
Interaction energies (kJ/mol)	E_1^1 -14.95	-15.64	E_1^1 -50.89
	E_1^2 -5.70	-12.30	-48.51
	E_2 -4.07	-4.64	-0.40
	E_3 -0.81	+1.75	-39.89
	E_4 -1.40	+1.79	-35.81

E_1 : $\text{O}_{\text{H}_2\text{O}} - \text{H}_{\text{HC,DMS}}\text{F}_{\text{FHC}} - \text{H}_{\text{H}_2\text{O}}$
 E_2 : $\text{O}_{\text{H}_2\text{O}} - \text{mol}_{\text{HC,DMS}}$
 E_3, E_4 : $\text{H}_{\text{H}_2\text{O}} - \text{mol}_{\text{HC,DMS,FHC}}$

Interaction Forms

Bond length (nm)	(F—H) 0.307	(O—H) 0.267
Interaction energies (kJ/mol)	E_1^1 -10.28	E_1^1 -9.60
	E_1^2 -13.62	-7.58
	E_2 -0.94	+2.41
	E_3 -0.32	-5.00
	E_4 -2.58	-2.07

E_1 : $\text{F}_{\text{FHC}} - \text{H}_{\text{H}_2\text{O}}, \text{O}_{\text{H}_2\text{O}} - \text{H}_{\text{DMS}}$
 E_2 : $\text{O}_{\text{H}_2\text{O}} - \text{mol}_{\text{DMS}}$
 E_3, E_4 : $\text{H}_{\text{H}_2\text{O}} - \text{mol}_{\text{FHC}}$

Heterogeneous coating

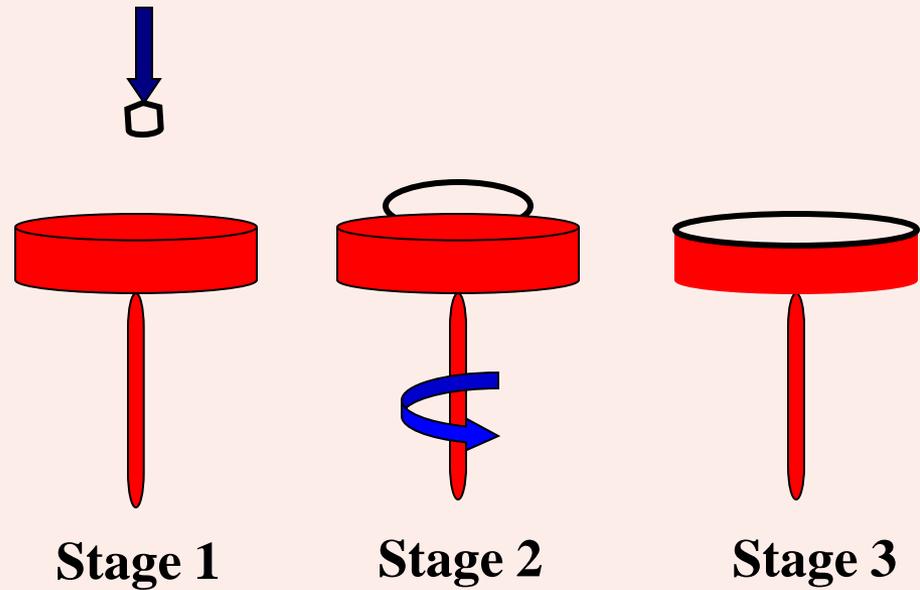
Objectives

- *Preparation and characterization of HC nanoparticle coatings in terms of hydrophobicity and icephobicity,*



Spin coating

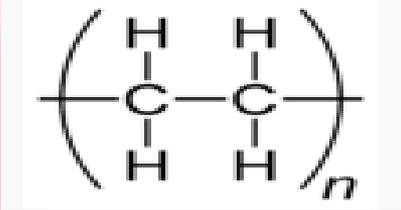
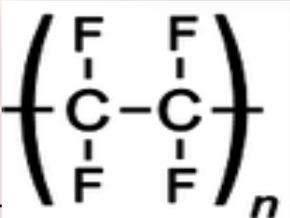
Laurel (*WS-400B-6NPP*)-CIGELE



spinning rate $\left\{ \begin{array}{l} V = 500 \text{ rpm} \\ T = 15 \text{ s} \end{array} \right.$

Methodology

□ *Preparation of heterogeneous nanoparticle coating*

Material	Structure
Polyethylene (PE)	
Polytetrafluoroethylene (PTFE)	

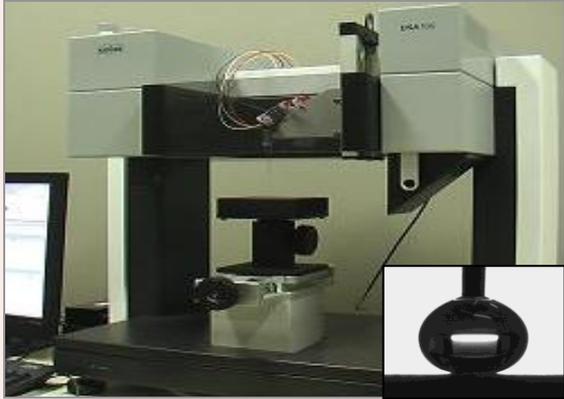
Methodology

➤ *Examples of “heterogeneous coating” preparation*

Material	Quantity	Solvent	Method	Company	Abbreviate
PE	1 gr	50 ml Toluene (at 110° cc)	Spin coating	Good-fellow	PE-spin
PTFE	1 gr	50 ml Toluene	Spin coating	Sigma- Aldrich	PTFE-spin
PE, PTFE	1 gr, 1 gr	50 ml, 50 ml Toluene	Spin coating	Good- fellow, Sigma- Aldrich	PE-PTFE
PE, PTFE	1 gr, 1 gr	100 ml Toluene	Spin coating	Good- fellow, Sigma- Aldrich	PE+PTFE

Characterization

Sample Wetting Properties



*Kruss DSA100 Water Contact Angle
Goniometer (CIGELE)*

Sample Anti-ice Performance



*Centrifuge adhesion test
machine (CIGELE)*

- *X-ray photoelectron spectroscopy (XPS)*
- *Scanning electron microscopy and energy-dispersive x-ray spectroscopy (SEM)/(EDS)*
- *Atomic force microscopy (AFM)*

Results: *HC nanoparticle coatings*

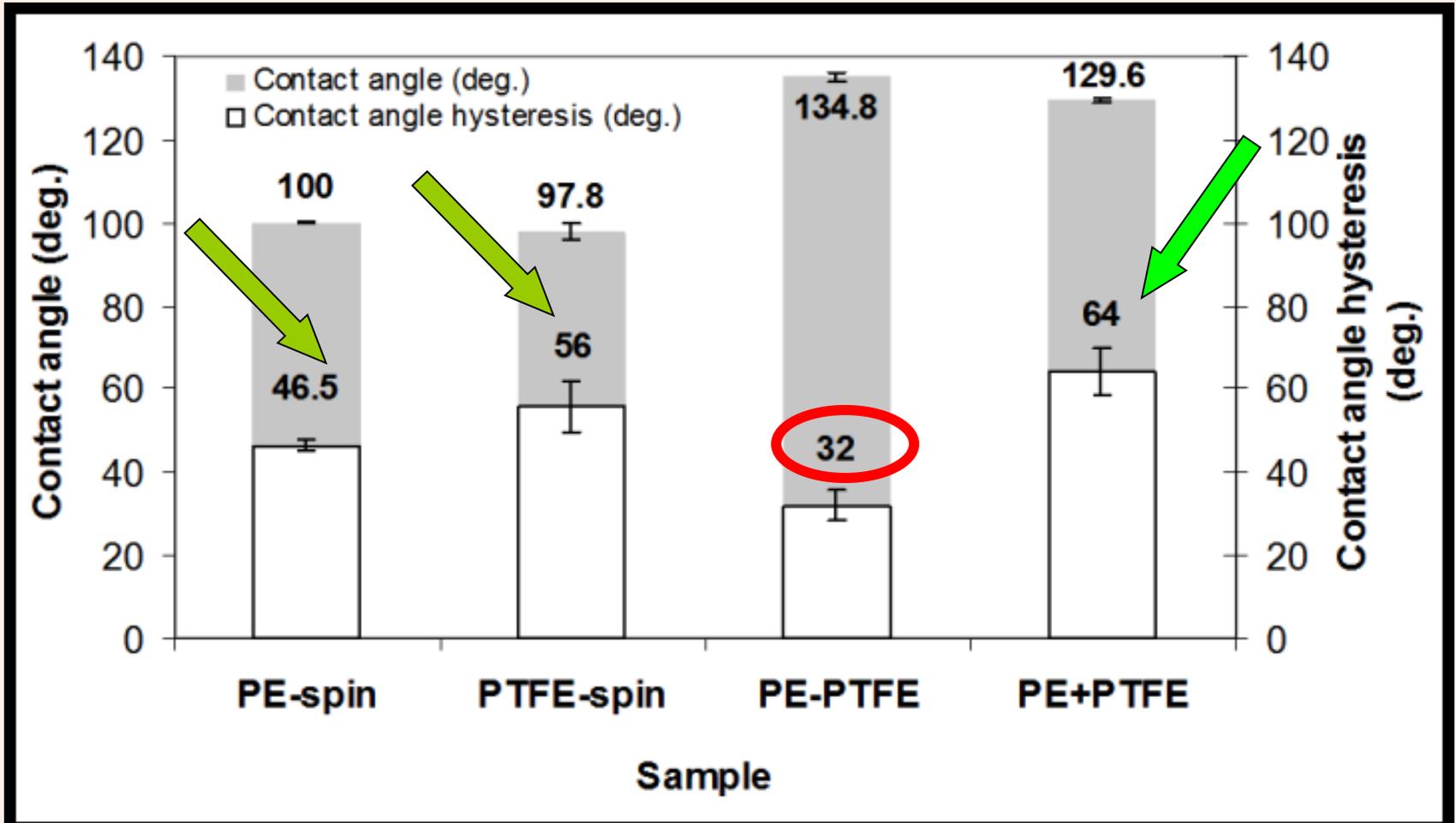
- CA values of homogeneous and HCs made of PE and PTFE



Coating	PE	PTFE (200nm)	PE-PTFE	PE+PTFE
CA values	100° ± 0.36	97.8° ± 1.83	134.8° ± 1	129.6° ± 0.6

- This table presents the ‘heterogeneity *effect*’, however there is problem of roughness effect created by PTFE nanocomposite.
- HC coating of PE-PTFE was prepared to investigate separately the hydrocarbon and fluorocarbon effect.
- Mixture of PE and PTFE, named as PE+PTFE coating, was also prepared to study the effect of roughness.

Results: *HC nanoparticle coatings*



Surface characterization: *AFM results*

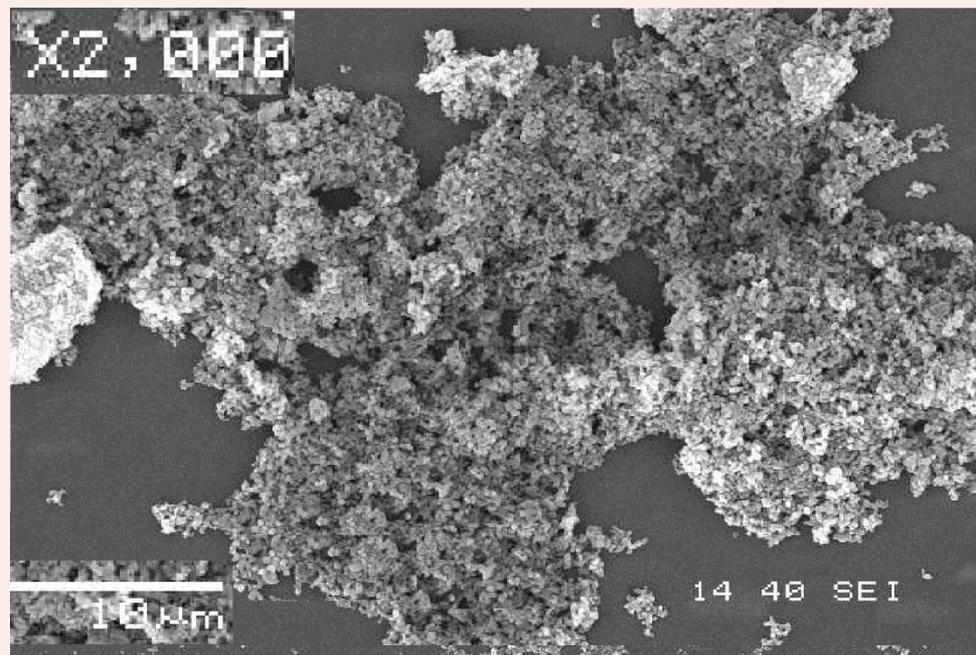
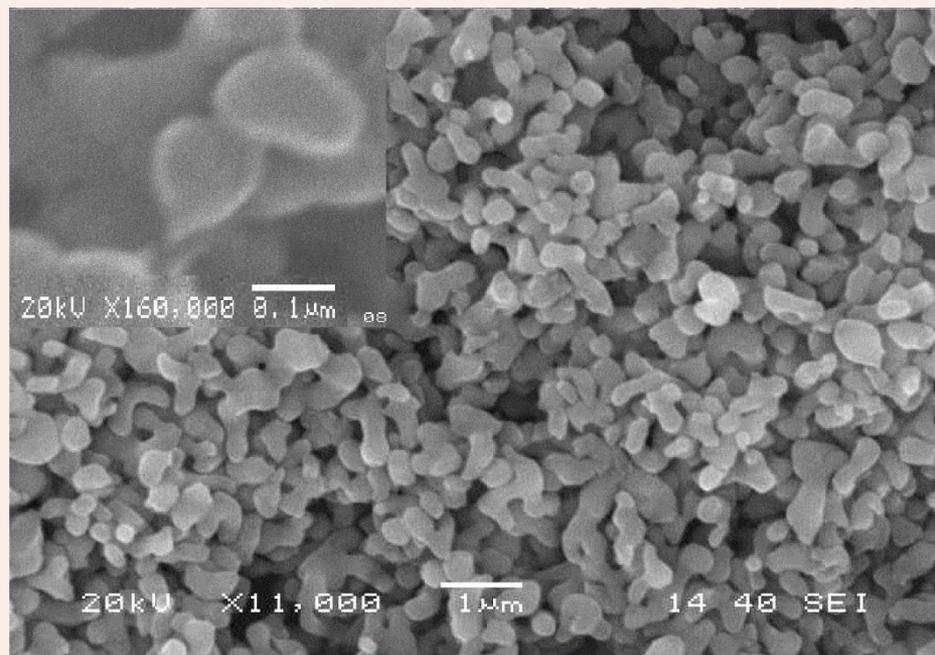
•The R_{rms} (nm) of homogeneous and HC samples.

Sample	Root mean square (nm)
PTFE-spin	165.5 ± 68.58
PE+PTFE	284.79 ± 173.14
PE-PTFE	239.85 ± 145

- The R_{rms} values of the PE+PTFE and PE-PTFE coatings are close together, however...
- The CA and CAH values of PE+PTFE sample were smaller and bigger, respectively, than what observed for PE-PTFE sample.

Surface characterization: *SEM results*

- SEM images of PE-PTFE sample, at $\times 30$, $\times 300$, $\times 2000$, $\times 11000$ and $\times 160000$ magnifications.



- SEM images of PE-PTFE demonstrates the propagated islands of nanoparticle in several areas and spots.

Ice adhesion results

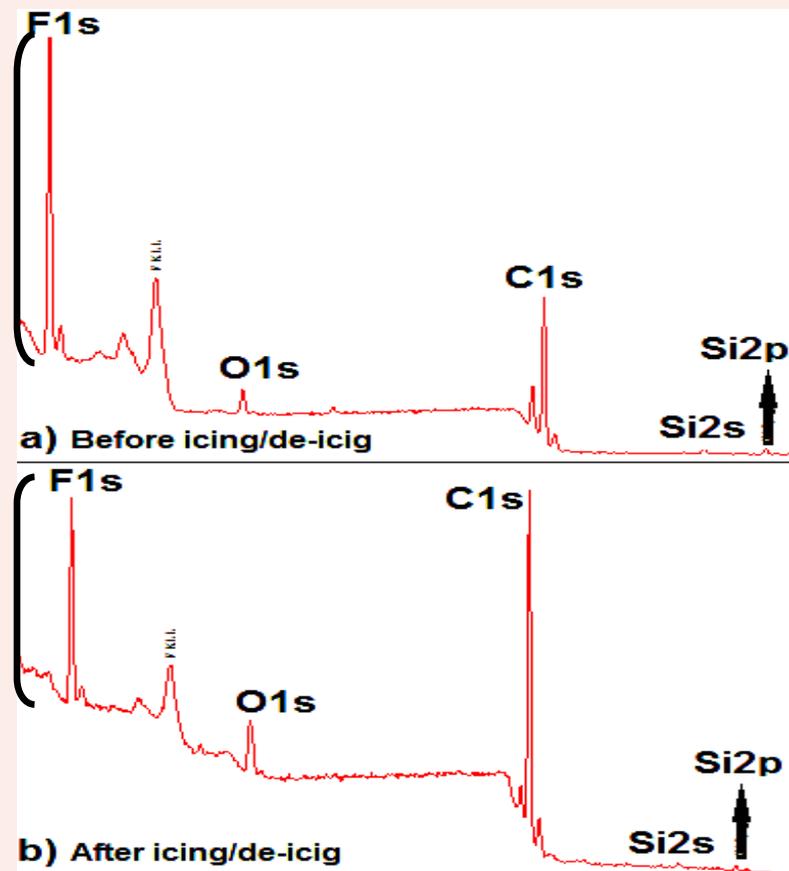
❑ Ice adhesion strength and *ARF* values of homo/heterogeneous nanoparticle coatings

Sample	Ice adhesion strength (kPa)	<i>ARF</i>
Polished Al	251.5 ± 27	1
PE-spin	220.8 ± 19.3	1.1
PE-PTFE	190.7 ± 34	1.32

➤ The *ARF* values of HC samples show at least ~1.3 times lower ice adhesion strength than those obtained on polished Al sample.

Surface characterization: XPS results

- XPS results of PE-PTFE sample showed partial remove of PTFE nanoparticles after icing/de-icing cycles.



XPS results of PE-PTFE sample before and after icing/de-icing

Element	C 1s %	F 1s %	O 1s %	Si 2p %
Before icing/de-icing	68.30	26.41	2.86	2.18
After 2 times icing/de-icing	83.37	10.10	5.26	1.28

Conclusions

-  The wetting behavior, morphology and ice-releasing performance of nanoparticle-based homogeneous and HC coatings on Al surfaces confirmed the “effect of heterogeneity” on sample hydrophobic and ice-phobic properties;
-  Meanwhile, CA and CAH measurements approved again the effect of heterogeneity on such coated surfaces;
-  The AFM analysis also confirmed different sample morphology due to heterogeneity effect on Al substrates;
-  Anti-ice performance of prepared HC nanoparticle coating was at least ~1.3 times lower than polished Al sample while for homogeneous PE-spin coating the ARF was ~ 1.1 times lower than polished Al sample;
-  The XPS analysis showed partial remove of PTFE nanoparticles after several icing/de-icing cycles;

Thank you for your attention!