Skoltech
Skolkovo Institute of Science and Technology

Thesis Changes Log

Name of Candidate: Igor Usachev
PhD Program: Physics
Title of Thesis: Performance of Aircraft Anti-Icing Fluids on Different Substrates
Supervisor: Prof. Vladimir Drachev
Co-advisor: Dr. Viktor Grishaev

The thesis document includes the following changes in answer to the external review process.
Dear Jury Members, First and foremost, I would like to express my sincere gratitude for your invaluable comments and helpful suggestions. I truly appreciate them. I have thoroughly revised my thesis based on your questions and comments. Now, I am confident that the overall quality and readability of my work have improved significantly. In response to your comments, I have provided a list of changes. The revised sections of my thesis are enclosed in quotation marks, and all modifications are highlighted in red. I look forward to seeing you at my defense. Kind regards, Igor Usachev
Revisions per suggestions of Prof. Vasily V. Vedeneev:
Comment 1: The structure of the thesis should be improved. The division into “Materials and Methods” and “Results” chapters makes the study hardly understandable. As a result, experimental and numerical parts of the study are messed in both chapters. It would be much more convenient to separate experimental part from the numerical part. **Response 1:** Thank you for the comment. The thesis follows the IMRaD (Introduction, Methods, Results, and Discussion) structure. Subsections are used to separate the experimental part

from the numerical part. To make the structure more clear, the last paragraph of the Introduction has been rewritten.

"Overall, this thesis is divided into five chapters. Chapter 2 provides a literature review on the objectives mentioned above. Chapter 3 describes the methods used in the work. Section 3.1 introduces the tested fluids and substrates, as well as the testing procedure itself. Section 3.2 explains the polarimetric method and the ice imaging method. Section 3.3 specifies the properties of the tested fluids, the mathematical model, and its numerical implementation. The results and discussion are provided in Chapter 4. Section 4.1 shows the results of ice contrast enhancement and automatic ice imaging. The influence of substrate properties on fluid freezing is discussed in Section 4.2. Section 4.3 presents the simulation results. Chapter 5 includes conclusive remarks and an outlook for future research."

Comment 2: More photographs of the experimental equipment and models are necessary: in the present state, the "Materials and methods" chapter has no pictures of what was tested, say, of produced surfaces.

Response 2: Thank you for pointing that out. I have added four figures and the following lines to Sections 3.1.2 and 3.1.3 to clarify this point:

"Figure 3.2 shows the prepared aluminum substrates. Figure 3.3 displays the prepared acrylic substrates.

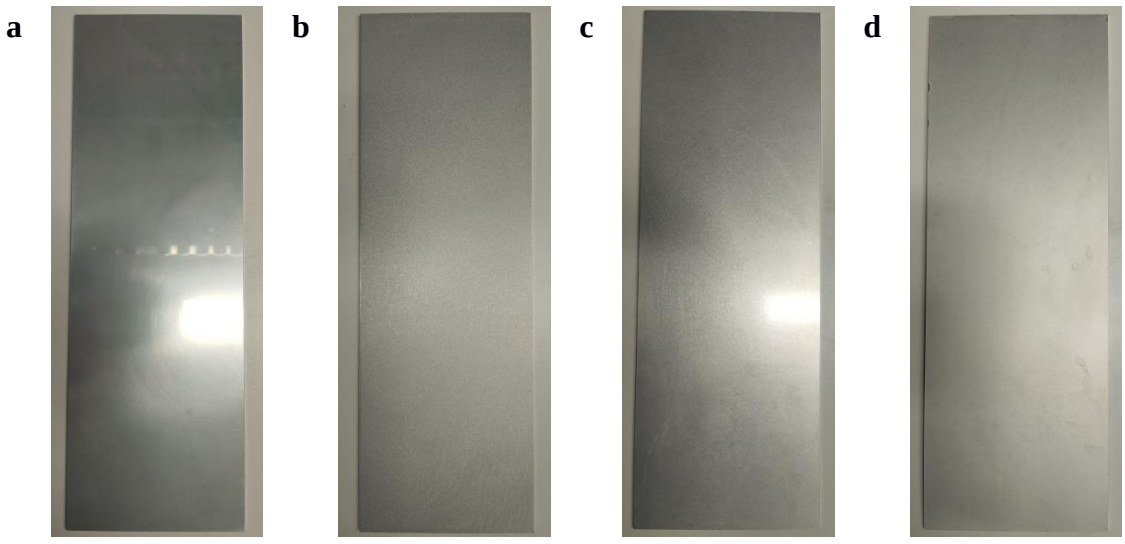

Figure 3.2 - Prepared aluminum substrates: a) Superhydrophilic, b) Rough, c) Glossy, d) Matt

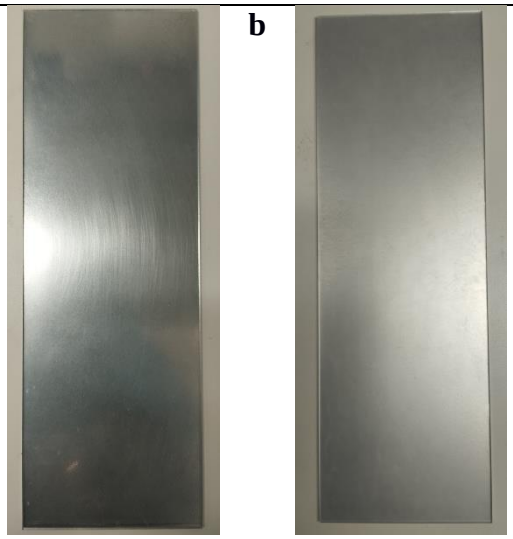

Figure 3.3 - Prepared acrylic substrates: a) Glossy, b) Matt

…

… Figure 3.8 shows a schematic of the test chamber with an ice imaging system. A picture of the test chamber is shown in Figure 3.9.

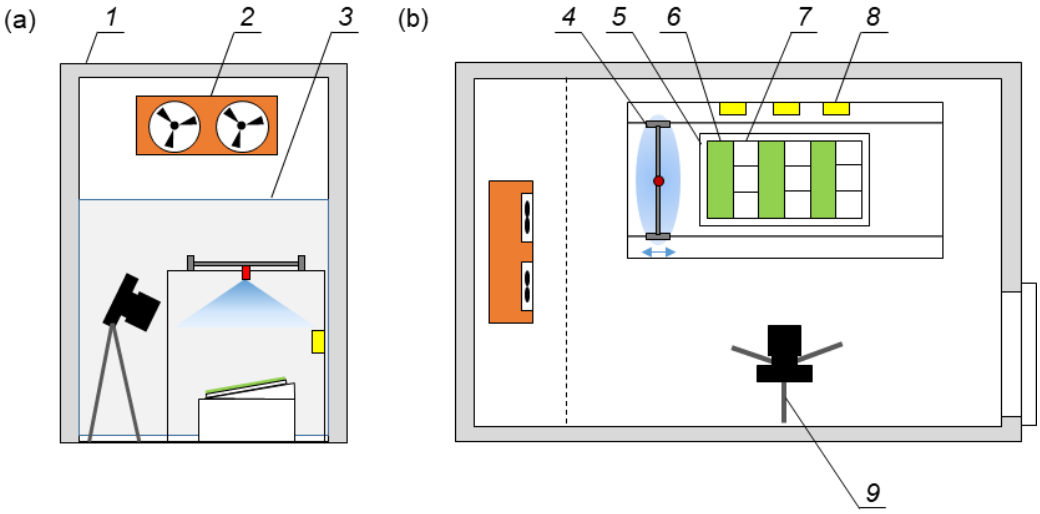

Figure 3.8 – Schematic of the test chamber with the ice imaging system: side view (a) and top view (b). 1 – cold chamber; 2 – heat exchanger; 3 – curtain; 4 – frame with a moving nozzle; 5 – freezing table; 6 – aluminum plates treated with an anti-icing fluid; 7 – ice catch panels; 8 – LED spotlights with polarizers; 9 – DSLR camera with an analyzer on a tripod.

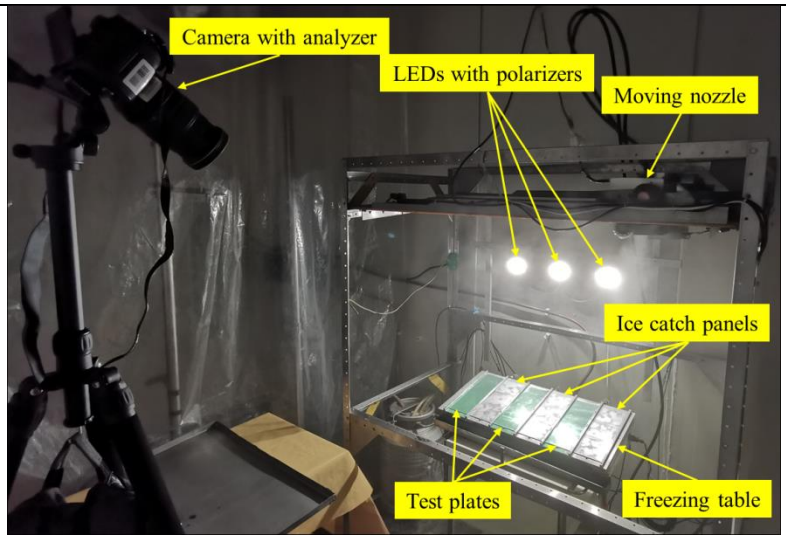

Figure 3.9 – Experimental setup."

Comment 3: Discussion of the numerical model starts abruptly in the experimental section 3.3.2 without any introductory words.

Response 3: Thank you for the comment. Introductory words have been added after Figure 3.22. Please note that the figure numbering has been changed.

"To calculate the freezing of a film, it is necessary to know how its thickness changes over time. Then, a velocity distribution is needed to calculate these changes. Let us use the Navier-Stokes equations for an incompressible viscous fluid and laminar flow to derive the velocity distribution."

Comment 4: I recommend to separate mathematical model and its numerical solution methods.

Response 4: Thank you for the comment. The mathematical model and its discretization have been separated in Chapter 3.

Comment 5: The numerical convergence is considered at the end of the thesis, after the results were presented. Meanwhile, it is a common practice to prove the reliability of the model through the convergence study first, and then to provide the calculation results. The structure of the form "mathematical model – numerical discretization and solution – verification (convergence study) – results" would be much more convenient and straightforward.

Response 5: Thank you for pointing that out. The model uses a hyperparameter called the stable layer thickness, which is taken from the results. Therefore, the results are presented first, and then a convergence study is conducted based on the thickness calculated from these results.

Comment 6: I could not find any validation of the program processing images to get iced surface region. Also, no justification of the image binarization threshold value.

Response 6: Thank you for the comment. Currently, icing is detected during the WSET by visual and tactile inspections. Therefore, naked eye accuracy is the main source of validation. For instance, Gagnon et al. compared their fluid failure determination algorithms to the average of three technicians using the same images as the algorithms [1].

Our model is first calibrated on a single image to best match human judgment by selecting a threshold value. The same quality of ice imaging in other images serves as validation for the model. The following text has been updated to include information about model validation (page 38):

"… ~~At~~ Third, the image is transformed into a binary image ~~one~~ by applying a ~~the~~ threshold to match ~~coincide~~ the output results with ~~the~~ visual and tactile inspections. The threshold is selected based on a single image to best match human judgment. The same quality of ice imaging in other images serves as validation for the model. In the same way, Gagnon et al. used the average opinion of three technicians to validate their fluid failure determination algorithms [42]."

Comment 7: Where equations (3.2) came from? If it's interpolation of experimental data, what is its accuracy (dispersion)?

Response 7: Yes, these are the results of interpolation. The text has been updated to include information about the root mean squared errors of the fitting process (after Eq. 3.1b). Please note that the equation numbering has been changed.

"*The root mean squared errors for the fitted viscosities of SAE Type II and Type IV fluids are 8.187 Pa·s and 7.926 Pa·s, respectively.*"

Comment 8: What happens with the topology of the fluid domain when the fluid thickness=0?

Response 8: Thank you for the question. In my simulation, the thickness of the film never reaches zero. The film drains for a while and then freezes. Therefore, the topology does not change.

Comment 9: What is "zero porosity" condition and why is it valid?

Response 9: Thank you for the question. This means the zero flux condition. That is, the flux at the boundary is zero. This is valid, because the aluminum surface is impermeable to both anti-icing fluid and water. The following text has been updated (page 53):

"The boundary conditions are zero flux ~~porosity~~ at the plate surface $\left(\left.\frac{\partial C}{\partial y}\right|_{x,y=0,t} = 0\right)$ and water flux at the air/fluid interface $\left(\left.\frac{\partial C}{\partial y}\right|_{x,y=h,t} = -\frac{m''}{\rho_w D} C\right)$."

Comment 10: What is m″ in the interface condition and how is it calculated?

Response 10: Thank you for the question. The precipitation intensity is denoted as m″. This represents the average mass water flux of a spray system. It should correspond to 5 g/dm^2/h under WSET conditions [2]. I have added the following remarks after Eq. 3.4 to clarify this point:

"Here, m'' is the mass flux of water (5 g/dm^2/h under WSET conditions), and h is the thickness of the film."

Comment 11: How are the freezing and its propagation front calculated?

Response 11: Thank you for the question. The following changes have been made to the text to avoid misunderstandings:

"The discretized version of Eq. 3.7 at some time step at a node (i,j) takes the form:

$$\varphi_{i,j} = max\left(0, min\left(1, \frac{H^*_{i,j} - c_p T_{cr}\left(C^*_{i,j}\right)}{L_f}\right)\right). \quad (3.22)$$

Complete freezing in the node (i,j) is defined as when the mass liquid fraction is zero $(\varphi_{ij} = 0)$. The freezing front is located between the nodes where the mass liquid fraction is zero $(\varphi_{ij} = 0)$ and one $(\varphi_{ij} = 1)$. …"

Comment 12: How does the mathematical model include the surface condition (hydrophilic, hydrophobic, rough/smooth)? As far as I can see, they are not reflected in the model.

Response 12: Thank you for the question. The proposed model is designed for a smooth, hydrophilic aluminum surface. Expanding the model to other surfaces is a topic for future work. The following changes have been made to the text (page 87) to avoid misunderstandings:

"Therefore, the model is convergent and can be used to predict the fluid failure and icing kinetics of SAE Type II and IV fluids. Fluid manufacturers can implement the proposed model to check various combinations of fluid properties for the best anti-icing effect. The proposed model is designed for a smooth, hydrophilic aluminum surface. In the future, it is recommended to expand ~~validate~~ the model to include ~~for~~ SAE Type I fluid and substrates other than aluminum. …"

Comment 13: The results are published in three high-quality journal papers. However, each

paper has 10 co-authors from three different scientific centers so that the actual role of the PhD candidate in those papers and in the present study is questionable for this reviewer. I believe this question must be addressed in the thesis.

Response 13: Thank you for the comment. I took part in the experiments in a test chamber that formed the basis for all three published papers. I analyzed and visualized the experimental results. My special role in the second paper was to develop a program for automated ice imaging. In addition, I developed a numerical model and drafted our fourth paper. I wrote the dissertation based on the results obtained during my research. This information has been included in the introduction (page 17) to avoid misunderstandings.

"My personal contribution: I participated in experiments conducted in a test chamber. I analyzed and visualized the results of these experiments. Additionally, I developed a program for automated ice imaging and a numerical model, and verified them against experimental data."

Minor comments

Comment 14: Table 3.1: how the viscosity and surface tension were measured? Viscosity of Type II, IV fluids depends on the shear strain since they are non-Newtonian so that "viscosity" is not just a number. How should one interpret the "viscosity" given in that table?

Response 14: Thank you for the comment. The viscosity and surface tension values in the table are presented to show the relative difference among tested fluids. The dynamic viscosity in Table 3.1 was measured using an Ametek Brookfield DV2T at 20°C. The viscosity of the Newtonian SAE Type I fluid was measured using a spindle #18 at 70 rpm. The viscosities of SAE Type II and IV pseudo-plastic fluids were measured using a LV1 spindle at 0.3 rpm. The surface tension was measured by the pendant drop method using a Krüss DSA30S at 23°C.

I have included this information in the text (page 24) to avoid misunderstandings.

"**…** The properties of the fluids are presented in Table 3.1. The fluid densities were provided by the manufacturer. The dynamic viscosity was measured using an Ametek Brookfield DV2T at 20°C. The viscosity of the Newtonian SAE Type I fluid was measured using a spindle #18 at 70 rpm. The viscosities of SAE Type II and IV pseudo-plastic fluids were measured using a LV1 spindle at 0.3 rpm.

The surface tension was measured by the pendant drop method using a Krüss DSA30S at 23°C. First, a pendant drop with a volume of ~10 µl was formed on a needle with a blunt tip and an outer diameter of 1.83 mm. It took 10 seconds for the vibrations in the drop to die out. Then, its silhouette was recorded every second for 10 minutes to calculate the surface tension. Table 3.1 shows the average surface tension values based on three measurements."

Comment 15: Table 3.2: why there are no data on raw surface, without varnish or degasation?

Response 15: Thank you for the question. We used superhydrophilic aluminum substrates as the lower limit of wettability. The raw surface would not have affected our results, as its wettability was within the observed range. However, it would be better to use the raw plates as a reference point. The following text has been updated (page 25):

"In order to check the whole wettability range, extreme but available levels were prepared, such as a superhydrophilic and two kinds of hydrophobic aluminum surfaces. The raw surface would not have affected our results, as its wettability was within the observed range. …"

Comment 16: Figure 3.3: What is "RCA" in the plots?

Response 16: Thank you for the comment. RCA stands for receding contact angle. The arrow shows the maximum value of the drifting angles. The corresponding changes were added to the figure caption. Please note that the figure numbering has been changed.

"Figure 3.6~~3.3~~ – Dynamic contact angles of sessile water droplets on hydrophobic glossy and matt surfaces after the aging by the anti-icing fluids. RCA stands for receding contact angle. The arrow shows the maximum value of the drifting angles."

Comment 17: "The comparison of Villeneuve's results and our results is presented in Figure 4.6." However, there is no comparison in that figure.

Response 17: Thank you for pointing that out. I have added this figure to the text. Please note that the figure numbering has been changed.

"… The comparison of Villeneuve's results [41] and our results is presented in Figure 4.6.

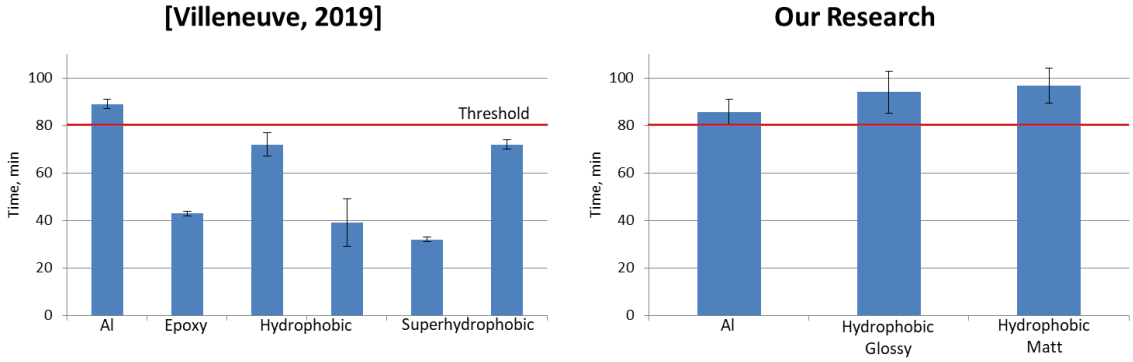

Figure 4.6 – Comparison of Villeneuve's results [41] with our own."

Revisions per suggestions of Prof. Carlo Antonini

Comment 1: Considering that the publications results match with the thesis content, it is thus hard for me to understand the individual role of the candidate in the research activities, first in the manuscripts, and consequently in writing the thesis.

Response 1: Thank you for your comment. I took part in the experiments in a test chamber that formed the basis for all three published papers. I analyzed and visualized the experimental results. My special role in the second paper was to develop a program for automated ice imaging. In addition, I developed a numerical model and drafted our fourth paper. I wrote the dissertation based on the results obtained during my research. This information has been included in the introduction (page 17) to avoid misunderstandings.

"My personal contribution: I participated in experiments conducted in a test chamber. I analyzed and visualized the results of these experiments. Additionally, I developed a program for automated ice imaging and a numerical model, and verified them against experimental data."

Major issues:

Comment 2: Table 3.1: is this data from literature or a result from this study? In the first case, please provide references. In the second case, please include details on measurement.

Response 2: Thank you for your comment. The details have been included in the text (page 24).

"**…** The properties of the fluids are presented in Table 3.1. The fluid densities were provided by the manufacturer. The dynamic viscosity was measured using an Ametek Brookfield DV2T at 20°C. The viscosity of the Newtonian SAE Type I fluid was measured using a spindle #18 at 70 rpm. The viscosities of SAE Type II and IV pseudo-plastic fluids were measured using a LV1 spindle at 0.3 rpm.

The surface tension was measured by the pendant drop method using a Krüss DSA30S at 23°C. First, a pendant drop with a volume of ~10 µl was formed on a needle with a blunt tip and an outer diameter of 1.83 mm. It took 10 seconds for the vibrations in the drop to die out. Then, its silhouette was recorded every second for 10 minutes to calculate the surface tension. Table 3.1 shows the average surface tension values based on three measurements."

Comment 3: Please provide standard deviations for data in Table 3.5 and 3.6, and Figure 3.10 and 3.11. Also, make sure you report only significant digits in Table 3.5 and 3.6.

Response 3: Thank you for pointing that out. The viscosity values in Tables 3.5 and 3.6

were measured once at −5°C, while the fluid was being heated at room temperature. Therefore, there are no standard deviations. This fact is mentioned in the text (page 43):

"Next, the beaker with the cooled fluid was placed in the rheometer at room temperature. The device was set at a velocity gradient from the working range (from 0.01 to 1 s^{-1}). The fluid temperature was continuously monitored with a thermocouple. The measurement continued for at least 5 minutes before the test temperature of –5°C was reached in the fluid. Measured viscosities and corresponding temperatures were recorded. Then, the procedure was repeated for each remaining velocity gradient."

Comment 4: Figure 3.12: why is viscosity considered constant for values of velocity gradient < 10^{-2} s^{-1}? Please clarify.

Response 4: Thank you for the comment. The viscosity at velocity gradient values below 10^{-2} s^{-1} was not measurable with the rheometer used. Therefore, I needed an approximation to fit a continuous viscosity function. The easiest way was to approximate a constant value. Furthermore, when compared to a linear approximation, the difference remained insignificant. The relevant comments have been added to the text (page 43):

"**…** Finally, the empirical function of the velocity gradient was fitted to the averaged data. The viscosity at velocity gradient values below 10^{-2} s^{-1} was not measurable with the rheometer used. The easiest way was to approximate a constant value. Therefore, the highest value for the viscosity was chosen as the value at the lowest measured velocity gradient. Furthermore, when compared to a linear approximation, the difference remained insignificant."

Comment 5: Page 56: it is claimed that the cooling time for a 25 um drop is only 17 ms. Based on a lumped mass assumptions, the characteristic time calculated as t* = rho*cp*V/ h*A = rho*cp*R/3*h can reach values of 17 ms for h > 1000 W/m^2K, which is a bit too high. Can the candidate please reconsider the estimation, to double check if 17 ms is realistic? Thank you.

Response 5: Thank you for such a great question. You performed the same calculations as I did at the beginning of model development. I found that for a water droplet $\left(D_{drop} = 20\ \mu m\right)$ in air $\left(h_{air} \approx 30.7 \frac{\mathrm{W}}{\mathrm{m^2 K}}\right)$, the time to cool down from 10°C to −5°C is:

$$t = \frac{c_{water}\ \rho_{water}\ V_{drop}}{h_{air}\ A_{drop}} \ln \frac{T_i - T_\infty}{T - T_\infty} = \frac{c_{water}\ \rho_{water}\ D_{drop}}{6\ h_{air}} \ln \frac{T_i - T_\infty}{T - T_\infty},$$

$$t = \frac{4200 \times 1000 \times 0.00002}{6 \times 30.7}\, ln \frac{10 - (-5)}{-4.95 - (-5)} \approx 2.6\ s.$$

However, the lumped capacitance method only works for solid bodies with negligible temperature gradients within them [3]. The thermal conductivity of water is three orders of magnitude lower than that of aluminum. Therefore, temperature gradients within water droplets cannot be ignored. Moreover, Hardy et al. [4] paid attention not only to convection, but also to evaporation effects, which are significant.

Comment 6: Page 71: "The results on superhydrophilic surfaces show that an increase in their roughness reduces the performance of the SAE Type I anti-icing fluid. This is different from hydrophobic surfaces, where an increase in roughness delays freezing (Figure 4.9)." In fact, looking at Figure 4.9, ice grows initially faster on superhydrophilic surface up to minute 8-9, but then ice accretion saturates later than on a hydrophilic surface. As such, how do the authors identify a performance reduction?

Response 6: Thank you for the comment. The fluid performance is evaluated based on the WSET criteria. This means we consider the initial surface freezing up to 10%. An increase in roughness supports the initial freezing of a rough superhydrophilic substrate (up to 45% in our case). Therefore, the performance is indeed reduced. The corresponding sentence (page 76) has been rewritten to avoid ambiguity:

"The results on superhydrophilic surfaces show that an increase in their roughness reduces the performance of the SAE Type I anti-icing fluid. This is different from hydrophobic surfaces, where freezing is delayed despite an increase in roughness (Figure 4.9)."

Minor issues:

Comment 7: Type I-IV de-icing fluids should be defined in Chapter 1. If the definition is provided in a later chapter (currently on page 23), indication should be given in Chapter 1 when first introducing the Type I-IV de-icing fluids (e.g. "see definition in Chapter X, Section Y).

Response 7: Thank you for pointing that out. The indication has been given in Chapter 1:

"**…** These types differ in composition, color, and minimum protective time. For more information, please refer to Chapter 3, Section 3.1.1."

Comment 8: Please be consistent in terminology, e.g. in the first chapter: first objective, second aim, third objective.

Response 8: Thank you for pointing out this inconsistency. I have fixed it.

"**…** Therefore, the second objective ~~aim~~ of the thesis is to develop a high contrast and automatic method to detect icing areas during the SAE tests."

Comment 9: Page 28: Advancing and receding contact angles are not, strictly speaking, dynamic contact angles. They are measured in quasi-static conditions. Please correct. Also, the correct unit is “ml/min” (small “m”).

Response 9: Thank you for the comment. Advancing contact angles are dynamic, as they are measured by the movement of a contact line. In contrast, receding contact angles of the hydrophobic surfaces drifted after being aged with anti-icing fluids. Therefore, we took their maximum values, which are actually quasi-static. Therefore, the word “dynamic” has been removed to avoid ambiguity. The typo with “ml” has also been fixed (page 29).

“To determine wettability, we measured advancing and receding ~~dynamic~~ contact angles of distilled water on obtained surfaces using the drop shape analyzer DSA30S (Krüss). Advancing contact angles were determined by moving the contact line of a sessile droplet during its volume increase from 4 ml to 44 m~~M~~l at a flow rate of 50 m~~M~~l/min. In turn, receding contact angles were registered during the droplet volume decrease from 44 m~~M~~l to 4 m~~M~~l at the same flow rate.”

Comment 10: Table 3.4: please check the significant digits for thermal diffusivity: only one?

Response 10: Thank you for pointing that out. I double-checked all the significant digits.

“Table 3.4 – Properties of solutions of SAE aircraft anti-icing fluids with water taken as constant.

Fluid Property	Type II	Type IV	Units
Density	1045	1032	kg/m^3
Specific heat	3498	3498	J/kg·K
Thermal conductivity	0.383~~0.38~~	0.383~~0.38~~	W/m·K
Thermal diffusivity	$1.05 \cdot 10^{-7}$ ~~0.0000001~~	$1.06 \cdot 10^{-7}$ ~~0.0000001~~	m^2/s

”

Comment 11: Figure 3.10 there is only one label for data on y-axis. Please have at least two or three (e.g. as in Figure 11).

Response 11: Thank you. I have corrected it. Please note that the figure numbering has been changed.

“

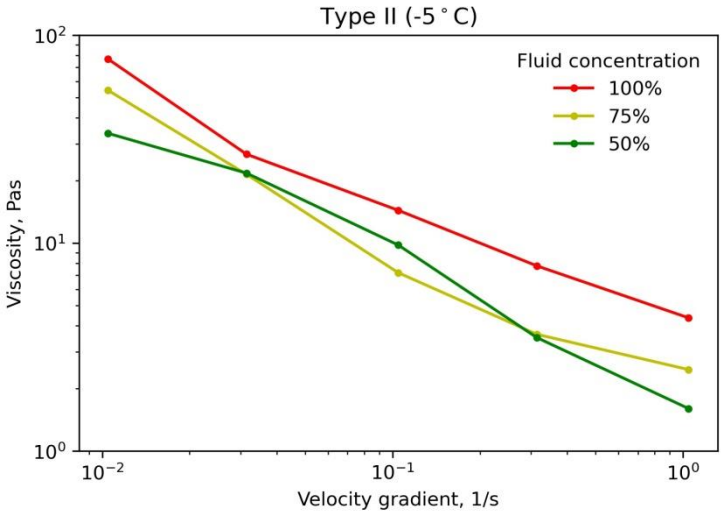

Figure 3.15~~3.10~~ – Type II fluid viscosity versus velocity gradient. All values are on a logarithmic scale.”

Revisions per suggestions of Dr. Andrey Glushchuk

Comment 1: All objectives of the presented research should be better formulated in the chapter 1.

Response 1: Thank you for the comment. A summary of all the objectives has been added at the end of the introduction (page 17).

“Considering the described issues, the objectives of this thesis are: *to conduct a comparative study on how substrate wettability, thermal conductivity, and roughness affect fluid performance; to develop a high-contrast and automatic method for detecting icing areas during SAE tests;* and *to develop a mathematical model of fluid freezing*.”

Comment 2: A schematic of the utilized climatic chamber should be presented properly in the section 3.1.3.

Response 2: Thank you for the comment. The schematic has been added (see Figure 3.8).

“… Figure 3.8 shows a schematic of the test chamber with an ice imaging system. A picture of the test chamber is shown in Figure 3.9.

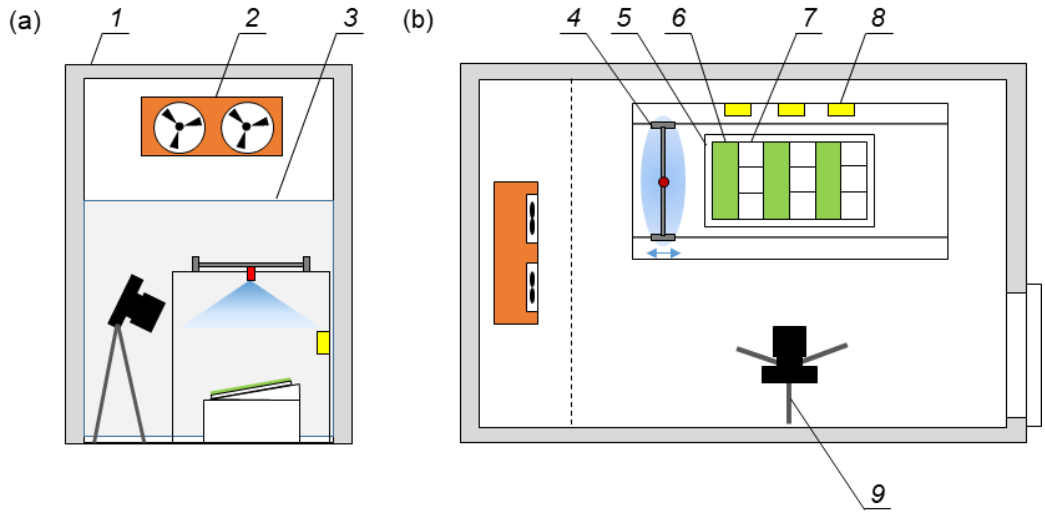

Figure 3.8 – Schematic of the test chamber with the ice imaging system: side view (a) and top view (b). 1 – cold chamber; 2 – heat exchanger; 3 – curtain; 4 – frame with a moving nozzle; 5 – freezing table; 6 – aluminum plates treated with an anti-icing fluid; 7 – ice catch panels; 8 – LED spotlights with polarizers; 9 – DSLR camera with an analyzer on a tripod."

Comment 3: A diagram of the realized numerical calculations should be introduced in the section 3.3.

Response 3: Thank you for pointing that out. The flowchart has been added to the end of the section 3.3.

"… Figure 3.23 shows a flowchart of numerical calculations performed.

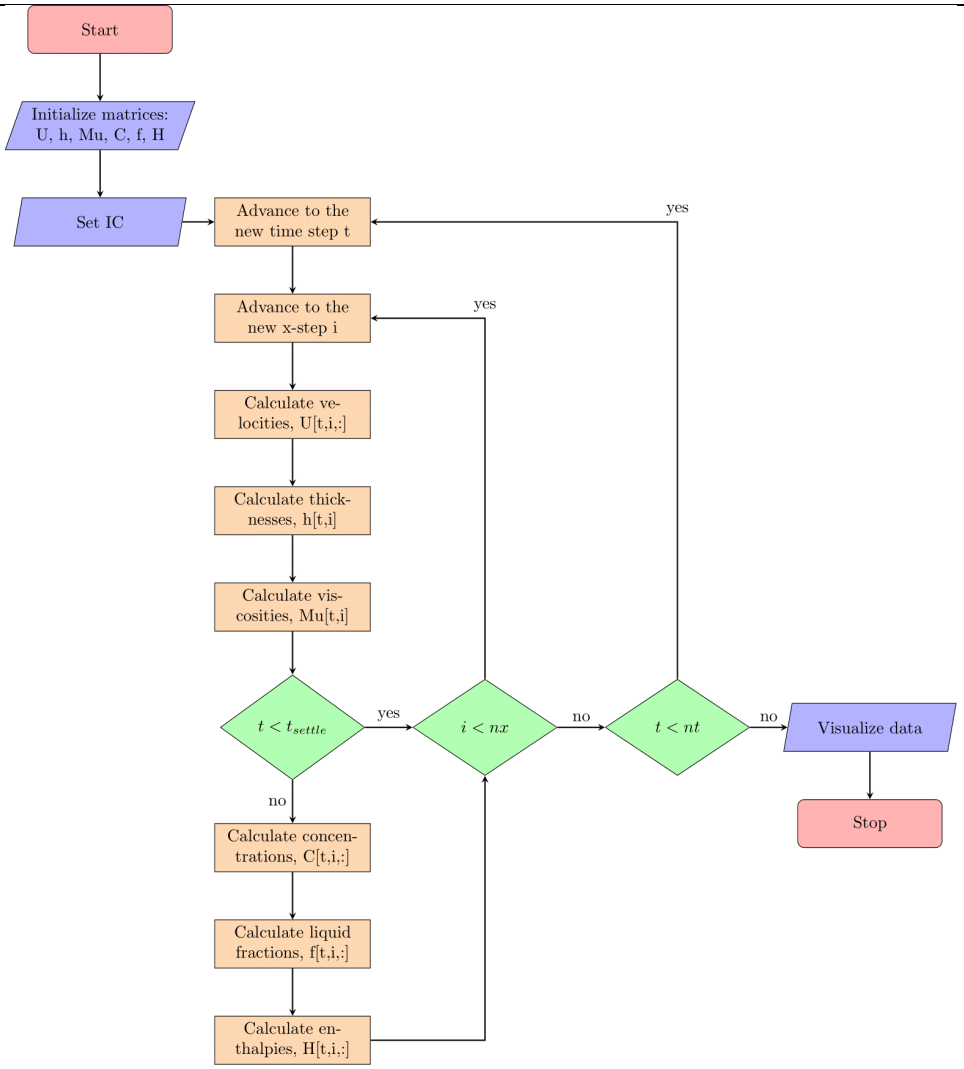

Figure 3.23 – Simulation flowchart.”

Revisions per suggestions of Prof. Carlo Saverio Iorio

Comment 1: Please, check the quality of Figure 3.14. Also, it would be nice to show at the beginning of the thesis some images showing the actual problem (ice on wings, de-icing systems).

Response 1: Thank you for the comment. The figure has been updated. Please note that the figure numbering has been changed.

“

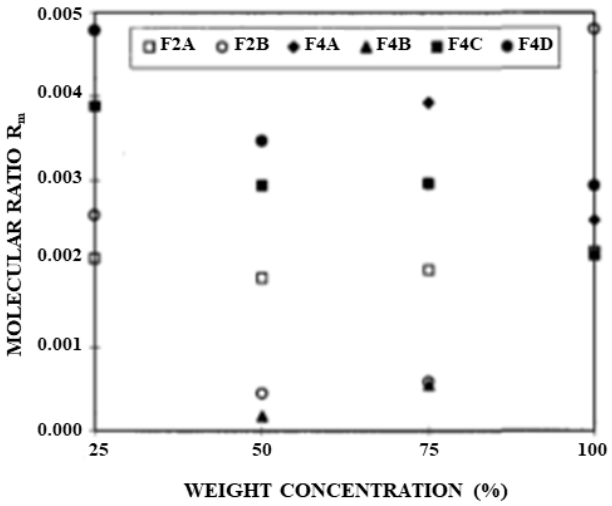

Figure 3.19~~3.14~~ – Type II and IV Molecular Ratio [54].”

A figure showing the anti-icing treatment of an aircraft has also been included at the beginning of the thesis.

“

Figure 3.1 – Anti-icing treatment [56]”

Comment 2: The polarimetric automatic detection gives impressive results. However, I could not find any estimation of the errors (like false positives/false negatives) that the method will provide. I understand that may be it is difficult at this point to evaluate them, but I would add a short paragraph discussing a possible reliability of the methodology.

Response 2: Thank you for pointing that out. Currently, icing is detected during the WSET by visual and tactile inspections. Therefore, naked eye accuracy is the main source of validation. For instance, Gagnon et al. compared their fluid failure determination algorithms to the average of three technicians using the same images as the algorithms [1].

Our model is first calibrated on a single image to best match human judgment by selecting a threshold value. The same quality of ice imaging in other images serves as validation for the model. The following text has been updated to include information about model validation (page 38):

"… ~~At~~ Third, the image is transformed into a binary image ~~one~~ by applying a ~~the~~ threshold to match ~~coincide~~ the output results with ~~the~~ visual and tactile inspections. The threshold is selected based on a single image to best match human judgment. The same quality of ice imaging in other images serves as validation for the model. In the same way, Gagnon et al. used the average opinion of three technicians to validate their fluid failure determination algorithms [42]."

Comment 3: It could also be valuable if a brief discussion will be added, concerning the polarimetric method, about its potential for detecting also ice thickness. Is it possible?

Response 3: The goal of anti-icing treatment is to remove any ice contaminants, regardless of their thickness [5]. Therefore, determining the ice thickness is not a major concern. However, there are ideas about how to determine ice thickness that can be found in literature. Pope et al. proposed a system comprising Raman spectroscopy used to detect key characteristics of ice formation on aircraft wings and engines in real time, capable to determine ice thickness [6]. Farzad proposed the Colour-Coded Point Projection (CCPP) method to determine ice thickness. The CCPP method is based on structured light triangulation [7].

But, the polarimetric method can only use the intensity of the reflected light on a camera sensor (pixel values). The method provides high pixel values for icing. The thickness of the ice layer can potentially influence this value. Unfortunately, the polarimetric method also highlights the structure of ice, which is invisible to the naked eye. This means that the pixel values for ice have large gradients due to the orientation of optical axes in ice crystals. These gradients make it impossible to take into account the influence of ice thickness, if any. The following paragraph has been added to the text (page 65) to clarify any misunderstandings.

"Furthermore, this method revealed the structure of ice crystals, which is invisible to the naked eye (Figures 4.3-4.4). The orientation of optical axes in ice crystals causes large gradients in the intensity of reflected light (pixel values). The thickness of ice can also affect the pixel values. Therefore, both the structure of ice crystals and their thickness affect the pixel values. However, the goal of anti-icing treatment is to remove any ice contaminants, regardless of their thickness [2]. Therefore, determining the ice thickness is beyond the scope of current research."

Revisions per suggestions of Prof. Oleg Vasilyev

Comment 1: The Abstract should be rewritten to provide a clear and comprehensive overview of the dissertation, including its goals, methodologies, and key findings. It should be logically organized and explicitly state what was studied, covering both the experimental and computational studies conducted. The abstract should also describe the experimental techniques developed and verified, and highlight the main findings. In other words, it should provide the reader with a solid understanding of the dissertation's content. Currently, this is not easily achieved in its present form. Additionally, the abstract should be written in the third-person perspective; the use of "we" is inappropriate in this context.

Response 1: Thank you pointing that out. The abstract has been thoroughly revised based on your feedback.

"The current PhD thesis focuses on the performance of aircraft de/anti-icing fluids on different substrates and the enhancement of endurance tests. These tests evaluate the performance of fluids and are conducted in accordance with the standards set by the Society of Automotive Engineers (SAE). ~~Some aircraft anti-icing fluids can dangerously reduce their performance on some composites and hydrophobic surfaces~~.

The first challenge addressed in this study is faster freezing of some fluids on composite materials and hydrophobic surfaces. These surfaces have properties such as wettability, thermal conductivity and roughness that differ from those of conventional aviation aluminum. Other challenges addressed in the work include the low ice contrast during testing and the time-consuming and subjective testing procedure. Moreover, it is not economically feasible to assess the performance of any fluid, especially during its development, solely through endurance tests.

The goals of the current research are to analyze the performance of aircraft de/anti-icing fluids SAE Types I, II and IV on substrates with different wettability, roughness and thermal conductivity, to image and predict freezing of the fluids. Therefore, ~~we considered~~ interaction of commercially available Newtonian and pseudo-plastic anti-icing fluids on different substrates was considered. In freezing drizzle simulations, no noticeable effect of surface wettability was observed on the performance of pseudo-plastic fluids. The difference between previous works can be caused by different properties of fluids and surfaces used in the work. Meanwhile, the Newtonian fluid took longer to freeze on rough hydrophobic surfaces. Separate experiments with rough and smooth surfaces demonstrated that roughness itself cannot delay the freezing of the Newtonian fluid.

It was also found that the decrease of substrate thermal conductivity promotes the freezing of Newtonian and low viscous non-Newtonian fluids compared to the thicker non-Newtonian one.

This finding shows it is crucial to find holdover times for the fluid SAE Type II on composites. ~~The difference between previous works can be caused by different properties of fluids and surfaces used in the work.~~ Furthermore, the physical adsorption of fluids in the surface texture leads to the drifting of receding contact angles of water on hydrophobic surfaces, thereby worsening their water-repelling properties. Thus, smooth hydrophobic surfaces are probably the best solution for ice mitigation systems contacting aircraft anti-icing fluids.

~~During endurance tests of aircraft de/anti-icing fluids, their failure is evaluated by a naked-eye visual inspection and manual tactile check. Such practice has issues with low ice contrast, is time-consuming, and subjective. Here we show how to enhance~~ The polarimetric method for enhancing ice contrast in the visible spectrum ~~by~~ using ice birefringence and polarized light reflection was demonstrated. The method can be used for both visual inspection and automatic ice imaging. Moreover, ~~it is not economically feasible to assess the performance of any fluid, especially during its development, solely through endurance tests. Therefore,~~ a fast and cheap continuum model of fluid freezing based on the Navier–Stokes equations, coupled with the diffusion and enthalpy equations, was developed and validated against experimental data. The discrepancy between the experimental and numerical results was about 10% once ice coverage surpassed 5% of the surfaces tested. The model can quickly monitor fluid performance before testing. It can also help in developing new types of fluids."

Comment 2: The Conclusion section currently resembles an abstract rather than a comprehensive discussion of the research findings. It should be revised to provide a more in-depth analysis, organized systematically and logically by categories, for readers who have already read the entire dissertation. For example, it should address which aspects or properties were studied, which were found to be more or less significant, and which had minimal influence. Given the comprehensive nature of this research, the conclusion should reflect this depth, with a clear and well-organized structure that is easy to read and follow.

Response 2: Thank you for the comment. The conclusion has been revised based on your feedback.

"… The obtained results can be divided into three parts. First, let us discuss how the performance of fluids is affected by the substrate. ~~Firstly, tests~~ Tests with different substrates have shown that changes in their wettability ~~change influences~~ influence only ~~on~~ the freezing of the AAF SAE Type I. All the differences for other SAE fluid types have remained within the standard deviation of experiments. *No dangerous decrease in endurance times is observed for hydrophobic coatings* used in this work. Discrepancies ~~Contradictions~~ with previous works ~~probably~~ depend on

the properties of the fluids and substrates used. Moreover, the use of *hydrophobic coatings provided a significant increase in the endurance times of the AAF SAE Type I*. Meanwhile, *surface aging of rough hydrophobic substrates by anti-icing fluids has led to the drifting of their receding contact angles*. ~~This means a decrease in the water-repelling properties of the substrates.~~ Therefore, the performance of a fluid depends on its compatibility with a specific substrate. ~~smooth hydrophobic surfaces can be applied as the best passive ice mitigation solution.~~

The surface wettability is also altered by its roughness. Experiments with the surface roughness of superhydrophilic samples have shown that *roughness alone can't delay a fluid failure*. But, an increase in the roughness of hydrophobic substrates leads to an increase in their endurance times. Therefore, *AAF SAE Type I performance depends on both surface wettability and roughness*.

Changes in thermal conductivity of substrates affect fluids' performance with a strong dependence on the SAE fluid type. The decrease in thermal conductivity of ~~a~~ substrates combined with the increase in their surface roughness provides faster freezing of AAFs SAE Type I and II. Currently, the holdover times for SAE Type I fluids are specified for composites (low thermal conductivity), in contrast to Type II fluids. Therefore, the holdover times for SAE Type II fluids should also be generated for composites due to ~~because of~~ their potential for faster icing. Meanwhile, the decrease in thermal conductivity of ~~a~~ substrates provides slower freezing of AAF SAE Type IV. Here, the holdover times calculated for aluminum also serve as a lower limit for composites. Since this work has been limited to fluids from a single manufacturer, a comparative study with fluids from other brands is necessary to understand how differences in their properties influence the observed effects.

~~Secondly, the~~ Second, let us discuss the enhancement of the WSET test. *The polarimetric method greatly improves ice contrast* for both visual observations and an automatic ice imaging system. It is explained by ice birefringence. Therefore, it is highly recommended to use the method as a primary tool for visual inspection in all endurance tests. *The proposed image processing algorithm allows determining the whole icing kinetics* during endurance tests. The optical method, combined with the image processing algorithm, can be employed in an automatic ice detection system for these tests.

Finally, the numerical model has been developed and verified using experimental data. The model's convergence has been demonstrated. *The model predicts surface freezing of AAFs SAE Types II and IV on aluminum substrates*. The model can check the performance of fluids before conducting expensive and time-consuming tests. However, the model needs to be extended to SAE Type I fluid and other substrates besides aluminum."

Comment 3: The notation *m″* is ambiguous; its intended meaning is mass flux density. A

clearer term would be either $\dot{m}$ or q_{m}. The notation $(\cdot)''$ may also be confusing, as it often implies a second derivative.

Response 3: Thank you pointing that out. The notation m'' is widely used in AMIL papers [8], [9], and also in some textbooks [3]. That is why I used it in my dissertation.

Comment 4: The free-surface condition $\left.\frac{\partial u}{\partial x}\right|_{x,y=h,t} = 0$ appears to be physically incorrect. It seems the author intended a stress-free condition at the surface, $\left.\frac{\partial u}{\partial y}\right|_{x,y=h,t} = 0$. This should be verified and corrected if it is a typographical error. If it is not a typo, then the numerical results presented in the dissertation are likely incorrect and will need correction.

Response 4: I sincerely appreciate your feedback. I definitely meant a stress-free condition $\left.\frac{\partial u}{\partial y}\right|_{x,y=h,t} = 0$. The typo has been corrected (page 52).

Comment 5: The symmetry conditions $\left.\frac{\partial u}{\partial x}\right|_{x=L,y,t} = 0$ at the computational domain boundary are never discussed. These or other boundary conditions applied in the simulation should be explicitly mentioned and discussed.

Response 5: Thank you for the comment. The condition $\left(u|_{x=0,y,t} = 0\right)$ has been added (page 52). Since we only have the first partial derivative of the velocity with respect to *x*, there is no need for a condition at the other end.

Comment 6: The boundary condition for solute concentration at the surface, $\left.\frac{\partial C}{\partial y}\right|_{x,y=h,t} = -\frac{m''}{\rho_{\mathrm{w}} D} C$, seems unphysical. The solute concentration should be determined bythe concentration of the precipitated fluid on the surface, which has a fixed concentration. Therefore, a Dirichlet boundary condition $C|_{x,y=h,t} = C_{\mathrm{p}}$, where C_{p} is the concentration of the precipitated fluid, would likely be more appropriate and physically accurate. The author should comment on this seemingly unphysical assumption and either justify it or correct it and redo the calculations.

Response 6: Thank you for the comment. Let us assume that the density of water is equal to that of a fluid ($\rho_{\mathrm{w}} \approx \rho_{\mathrm{f}}$). If we consider a thin control volume at the air-fluid interface (Figure 1), then we have a water flux through its upper boundary ($j_{\mathrm{in}} = -m'' C_{\mathrm{w0}}$) and the same flux through its lower boundary but after mixing with the fluid inside the volume ($j_{\mathrm{out}} = -m'' C_{\mathrm{w}}$). Moreover,

water diffusion is also observed through the lower boundary $\left(j_{\text{dif}} = -\rho_{\text{w}} D \frac{\partial C_{\text{w}}}{\partial y}\right)$.

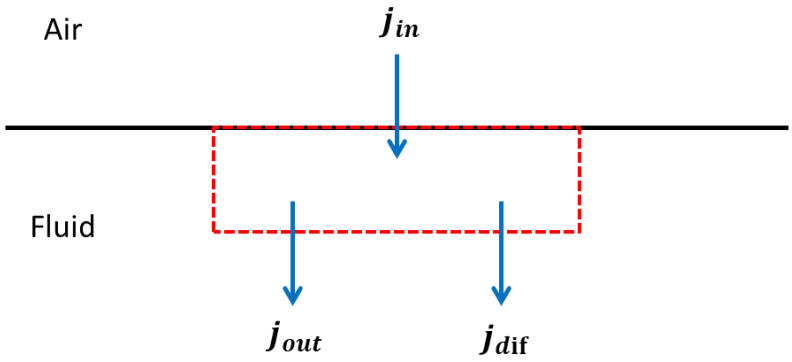

Figure 1: Air-fluid interface.

By applying conservation of mass, let us notice that:

$$j_{\text{dif}} + j_{\text{out}} - j_{\text{in}} = 0;$$

$$-\rho_{\text{w}} D \frac{\partial C_{\text{w}}}{\partial y} - m'' C_{\text{w}} + m'' C_{\text{w0}} = 0.$$

Accordingly,

$$\rho_{\text{w}} D \frac{\partial C_{\text{w}}}{\partial y} = m''(C_{\text{w0}} - C_{\text{w}});$$

$$\frac{\partial C_{\text{w}}}{\partial y} = \frac{m''}{\rho_{\text{w}} D}(C_{\text{w0}} - C_{\text{w}}).$$

Note that $C_{\text{w0}} = 1$ and $C_{\text{w}} + C_{\text{f}} = 1$. It follows that:

$$\frac{\partial(1 - C_{\text{f}})}{\partial y} = \frac{m''}{\rho_{\text{w}} D}\left(1 - (1 - C_{\text{f}})\right);$$

$$\frac{\partial C_{\text{f}}}{\partial y} = -\frac{m''}{\rho_{\text{w}} D} C_{\text{f}};$$

Therefore, we obtain the above-mentioned boundary condition.

Comment 7: The effect of roughness was only studied experimentally. I recommend adding a discussion on how to extend the simplified model to numerically incorporate surface roughness.

Response 7: Thank you for the comment. A brief discussion has been added to the text (page 52).

"The effect of surface roughness can be taken into account using the Navier slip condition [61]. The slip length, λ, typically represents wall slip. It is a fictitious distance below the surface where the velocity is zero. Most surfaces show very little slip, up to tens of nanometers, in contrast to rough superhydrophobic surfaces. Some measurements show slip lengths of a few tens of

microns for such surfaces, accompanied by laminar drag reduction up to 40% [62]. Hence, the influence of surface roughness on smooth hydrophilic and hydrophobic surfaces remains negligible and is beyond the scope of current research."

Minor suggestions:

Comment 8: References to figures are inconsistent throughout the dissertation, with both "Figure" and "Fig." used. The author should select one notation and use it consistently.

Response 8: Thank you for pointing out this inconsistency. I double-checked all the references.

Comment 9: The abbreviation "©" is not defined and should be clarified.

Response 9: Thank you for the comment. The typo has been corrected (page 48).

"Here, the glycol concentration is assumed to be equal to the AAF concentration (C) ~~©~~, which is reasonable in the considered temperature range (private communications with the manufacturer)."

Comment 10: The dissertation requires reformatting due to excessive half-empty pages. New pages should only begin with a new chapter, while subsections should continue without page breaks.

Response 10: Thank you for pointing out this inconsistency. I have removed all excessive page breaks.

Comment 11: Many equations in the dissertation are numbered but never referenced. Equations that are not referenced in the text should not be numbered.

Response 11: Thank you for pointing out this inconsistency. I double-checked the numbering of all the equations.

References

[1] D. Gagnon, J.-D. Brassard, H. Ezzaidi, and C. Volat, "Computer-Assisted Aircraft Anti-Icing Fluids Endurance Time Determination," *Aerospace*, vol. 7, no. 4, p. 39, Apr. 2020.

[2] G-12ADF Aircraft Deicing Fluids, "AS5901C, Water Spray and High Humidity Endurance Test Methods for SAE AMS1424 and SAE AMS1428 Aircraft Deicing/Anti-icing Fluids." SAE International, 2014.

[3] T. L. Bergman, *Fundamentals of Heat and Mass Transfer*, 7th ed. Wiley, 2011.

[4] J. K. Hardy, "Evaporation of Drops of Liquid," 1947.

[5] "Manual of Aircraft Ground De-icing/Anti-icing Operations (Doc 9640-AN/940)." ICAO, 2000.

[6] J. Pope, D. Buttry, and A. R. Toews, "Detection of aircraft icing," US8325338B1.

[7] M. Farzad, "Experimental Study of Rivulet/ Ice Formation by Colour-Coded Point Projection Method," Concordia University, 2019.

[8] T. V. K. Tông, P. R. Louchez, and A. Zouzou, "Introductory analysis of draining and freezing of de-icing fluids," *Cold Reg. Sci. Technol.*, vol. 25, no. 3, pp. 207–214, Apr. 1997.

[9] A. Zouzou, P. Louchez, and S. Bernardin, "Modeling of the draining and freezing of aircraft de-icing and anti-icing fluids," in *36th AIAA Aerospace Sciences Meeting and Exhibit*, 1998, pp. 1–10.