Influence of Substrate Initial Temperature on Adhesion Strength of Ice on Aluminum Alloy

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Abstract

The present work investigates the influence of the initial temperature of a substrate on the ice adhesion strength by analyzing the freezing characteristics of water droplets adhered to the substrate. The ice adhesion strength on 6061 aluminum alloy was measured using a dedicated strength testing apparatus, and the freezing process of water droplets at different initial temperatures of the alloy surface was examined with a microscope. The results of the experiments show that the ice adhesion strength on the aluminum alloy surface at ambient temperature was twice as large as that measured on a colder surface (e.g., -5 °C). Combining the experimental results with the microscopic observation of the freezing process revealed that at high initial surface temperature (i.e. equal to 18 °C), the water droplets thoroughly spread on the aluminum alloy surface at high temperature, formed a larger contact area. In addition, the initial surface

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temperature would influence the type of crystallization. Moreover, the advantages and
disadvantages of thermal de-icing approaches, widely used in engineering (especially in
the high-speed rail and aerospace fields), were discussed.

**Keywords:** Thermal de-icing; Ice adhesion strength; Initial surface temperature;
Aluminum alloy; Contact interface

1. Introduction

In cold-climate regions, the inevitable accumulation of ice or wet snow on exposed
surfaces severely affects many industrial activities and causes potential hazards in
aircrafts, wind turbines, power lines, highways, and offshore platforms (Gohardani et al,
2013; He et al., 2017; Ryerson, 2011; Zhu et al, 2016). These issues may cause serious
socioeconomic problems, and many countries have been affected by ice accumulation:
for example, the collapse of power transmission lines in China owing to ice
accumulation caused huge economic losses (Ruan et al., 2016). In order to ensure safe
operation and efficient performance, accreted ice on a surface is usually removed by
active methods, which include mechanical scraping, heating de-icing and chemical
agents. However, the active methods suffer from various shortcomings, such as high
costs, huge energy consumption, pollution, etc. (Yue et al., 2016).

In recent years, novel passive methods, in which the wettability of a surface is
modified in order to improve its anti-icing ability, reduce the adhesion of water, prevent
ice formation on solid surfaces, or decrease the ice adhesion strength. A particular type
of passive method involves superhydrophobic surfaces (SHS), which are regarded as a
promising anti-icing solution. However, several studies have highlighted the limitations
of SHS, such as the poor durability of their anti-icing effect (e.g., Farhadi et al., 2011; Kulinich et al., 2015; Varanasi et al., 2010). Kulinich et al. (2011a, 2011b; 2015) found that the wettability and the anti-icing ability of SHS changed during repeated icing/de-icing cycles, owing to the deterioration of the microstructure.

Therefore, active methods are still widely used in practical applications to remove ice accumulated on solid surfaces, especially in the high-speed rail and aviation fields, where thermal de-icing is the method of choice to melt accreted ice.

The fundamental mechanism of freezing of suspended or free droplets has been the subject of extensive research over the past few decades. Various efforts have been made to develop theoretical or mathematical models of droplet freezing and to validate them experimentally (e.g. Chaudhary et al., 2014; Fumoto et al., 2012). McDonald et al. (2017) studied the crystallization of a droplet on super-cooled hydrophobic surfaces and measured the ice adhesion strength, which was essential for understanding the influence of the surface characteristics on the freezing process and developing a method to measure the ice adhesion strength on SHS.

In the cold regions of China, water or snow near the rails frequently attaches to the train chassis during high-speed rail operation and freezes into ice with serious consequence for the safety, comfort, and service life of the key components. In order to ensure safe operation, along with optimal performance and speed, the accreted ice on the chassis is usually removed using thermal methods. However, owing to the large temperature difference between the chassis surface and its surroundings, after removing the accreted ice by thermal methods, the melted ice may be frozen again. Furthermore,
it is the timing of de-icing that is critical to the thermal de-icing method.

Taking into account the large temperature difference after adopting thermal de-icing methods, the objective of the present work is to investigate the influence of the initial surface temperature on the ice adhesion strength on aluminum alloy. The present paper analyzes the mechanism depending on the freezing process of water droplets on solid surface at different temperatures. In addition, the influence of the initial surface temperature on the energy needed for the application of the thermal de-icing method is discussed. The results of this study provide an improved reference for the implementation of conventional thermal approaches in de-icing technologies.

2. Test conditions

The ice adhesion strength was measured under different freezing conditions, and the adhesion strength values were then used to evaluate the influence of the initial surface temperatures on the stability of the accreted ice on the sample surface. The constantly changing morphology of water droplets or ice on the aluminum alloy during the freezing process was examined with a microscope.

2.1. Materials

Aluminum alloy widely used in the aerospace industry and other engineering fields (Ruan et al., 2016; Zuo et al., 2015). Therefore, in this work, 6061 aluminum alloy (40 \( \times \) 40 \( \times \) 2 mm\(^3\) in size) purchased from the Fushun Aluminum Co., Ltd., Liaoning (China) as the sample material. Ten microliters of water was dropped onto the sample surface using a micro pipette.
2.2. Temperature

Generally, ice formation takes place at low temperatures, ranging from -6 to -20 °C. This same range was also selected in many previous studies (e.g. Kermani et al., 2007). As the average winter temperature in China is below -20 °C, the final surface temperatures in the present study were respectively set to -5, -10, and -15 °C, which were considered as representative warm, medium, and cold environments.

The environmental conditions of the experiments were controlled using a climate chamber, in which the ambient temperature and humidity were maintained at approximately 18 °C, and 50%, respectively.

2.3. Experimental apparatuses

The ice adhesion strength was measured using a purposely-built apparatus to evaluate the effect of the initial surface temperature on the contact stability of ice. The freezing process of water droplets at different surface temperatures was recorded using a microscopic observation device.

2.3.1 Ice adhesion strength testing device

The measurement method of the adhesion strength referred the ASTM D3528 (2002) standard, and the purposely built apparatus to measure the ice adhesion strength was assembled and the measured procedures was detailed according to the ASTM D3528 (2002) standard and the study by Zou et al. (2007). As shown in Fig. 1, the key components of the apparatus were freezing unit, water-cooling unit, force-measuring
unit, along with a push-pull mechanism. The cooling unit contained a semiconductor cooling device to refrigerate the surface, whereas the force-measuring unit included a scraper mounted on a sliding table and a draft gauge. During the testing process, the scraper pushed the ice until it was completely peeled off and the maximum peeling force was recorded by the draft gauge. The measurement precision of the draft gauge is 0.01 N.

![Diagram of the testing device](image)


Fig. 1. Schematic illustration of tangential ice adhesion strength testing device.

Since this work is focused on the influence of the temperature on the ice adhesion strength, the subsequent tests were conducted only after the surface temperature of the sample had returned to a value close to ambient temperature (~18 °C). The temperature was measured by a K-type thermocouple located on the surface. A typical cooling curve from ambient temperature to a surface temperature of -5 °C, is shown in Fig. 2.
Fig. 2. Cooling curve of a sample, showing temperatures measured at the sample surface.

2.3.2 Freezing process observation device

The apparatus used to monitor the freezing process, shown in Fig. 3, consisted of a microscope, an image collection system, and a cooling station with a temperature controller. The cooling station could directly refrigerate the aluminum surface. The microscope, equipped with a charge-coupled device (CCD) camera, was used to dynamically inspect the droplet morphology and the freezing process. The multichannel temperature recorder could simultaneously record the ambient, sample surface and droplet temperatures. In order to analyze the relationship between temperature and surface morphology of ice, multiple cameras were employed to synchronously monitor different parameters during the freezing process.
Fig. 3. Microscopic device and experimental set up for monitoring the freezing process

2.4. Experimental conditions

2.4.1 Adhesion strength tests

The temperature at which the water droplet made contact with the sample surface was defined as the initial surface temperature of the aluminum alloy substrate. Two different experimental conditions were considered, labeled Experiment 1 and Experiment 2 in the following.

In the case of Experiment 1, the water droplet was placed on the aluminum alloy substrate before cooling, i.e., the initial surface temperature was close to the ambient temperature (18 °C). Then, the substrate and the water droplet were cooled down to the predetermined temperature and maintained at that temperature for 3 min. Finally, the ice adhesion strength was measured using the purposely-built device as shown in Fig. 1. Three final temperatures (-5, -10, and -15 °C) were considered in the Experiment 1.
In Experiment 2, the aluminum alloy substrate was first cooled down to a specific initial surface temperature (i.e., -5, -10, or -15 °C), and the droplet was placed on the sample for 3 min to cool. Then, the ice adhesion strength was measured by the device described above. As the temperature of substrate only showed a very small variation when the droplet was set in contact with the surface, this effect could be neglected. Hence, we considered the final temperature of the substrate was equal to its initial temperature.

In order to reduce experimental errors and random errors, each experiment was repeated at least 10 times. After each test, the residual ice on the sample surface was removed in an acetone ultrasonic bath for 5 min and in a deionized water ultrasonic bath for another 5 min. The samples were then dried in an oven at 60 °C.

2.4.2 Droplet freezing process

The microscopic observation of the freezing process aimed to understand the relationship between the initial surface temperature of the substrate and the freezing characteristics, based on the observed morphological variations and the measured ice adhesion strength. For this purpose, we recorded the morphology of water droplets located on the solid surface during the freezing process, under different freezing conditions.

3. Results

3.1 Ice adhesion strength
The ice adhesion strengths of frozen water droplets at different initial temperatures of the surface are summarized in Fig. 4. The results indicate that the adhesion strength increased with decreasing final temperature, and with an approximately linear dependence. The ice adhesion strength measured in Experiment 1 was about twice that obtained in Experiment 2.

When the final temperature of the substrate surface was reduced from -5 to -15 °C, the rate of increase in adhesion strength for Experiment 2 was lower than for Experiment 1. In particular, when the final temperature ranged from -10 to -15 °C, the peeling strength measured in Experiment 2 was significantly smaller than that obtained in Experiment 1.

![Fig. 4. Measured ice adhesion strength for different surface temperatures.](image)

3.2. Droplet morphology

In order to gain a better understanding of the relationship between initial surface temperature and ice adhesion strength, the freezing process of a single water droplet (10 μl) with a final temperature of -10 °C was inspected under two different cooling
conditions. The area of the contact interface was measured immediately after placing the droplet onto the aluminum surface. The initial height of the water droplet on the aluminum alloy and width of the contact interface zone were approximately 1.57 and 3.05 mm, respectively.

3.2.1 Experiment 1

Fig. 5 illustrates the time evolution of the appearance freezing water droplet under the ‘Experiment 1’ conditions. As shown in figure, the freezing process can be divided into five stages: initially, water droplet got muddy and lost its transparency; at the same time, the contact area between the droplet and the surface of aluminum alloy increased; then, phase change and solidification processes started, followed by freezing of the water droplet and changes in the surface morphology of ice (in agreement with Arianpour et al., 2013 and McDonald et al., 2017); finally, frost was formed on the outer-shell of ice (images 16 to 20 in Fig. 5).

Fig. 5. Images showing the time evolution of a water droplet freezing on the substrate under Experiment 1 conditions. h₀, L₀, were the initial height of the water droplet and width of contact interface between droplet or ice and aluminum alloy, respectively.
After being placed on the sample surface, the continuous cooling of the droplet resulted in its transparency decreasing from top to the bottom, with a simultaneous increase in height owing to the expanding contact area. Going from image 1 to 5 of Fig. 5, the maximum diameter of the water droplet on the 6061 aluminum alloy increased to 3.47 mm, i.e., 1.14 times larger than that of the initial state, while the contact area increased by 1.30 times (image 5 of Fig. 5).

As the freezing progressed, the water droplet fully changed into an opaque state and a solid-liquid interface emerged, dividing the water-ice mixture in a darker and a brighter regions. At the same time, the height of the solid-liquid mixture increased and approached the initial height of the droplet (images 6-11 in Fig. 5). Comparing images 11 and 5 in Fig. 5 reveals, no obvious changes in the contact area; however, the height of the water droplet changed and increased. Therefore, it can be concluded that a volume expansion occurred as the solid-liquid interface moved from the bottom to the top of the water droplet.

Owing to the constraint and propelling effect of the frozen liquid, the remaining water had to move upwards and frozen into a peak. A clear increase in the height of the frozen droplet could be observed remarkably going from image 12 to image 15 of Fig. 5, leading to a peach-like appearance. However, no changes in the contact interface area were observed.

Images 15-20 of Fig. 5 illustrate the frost formation on the outer shell of the frozen droplet. Frosting first appeared at the top of the “peach” shape and then gradually covered it. As the cooling continued, the thickness of the frost layer began to increase.
3.2.2 Experiment 2

The images in Fig. 6 illustrate the time evolution of a water droplet freezing on the Al-alloy substrate under the conditions of Experiment 2. In this case, the freezing process can be divided into four stages: the formation and displacement of the solid-liquid interface, shape change, freezing and frost formation.

Fig. 6. Images showing the time evolution of a water droplet freezing on the Al-alloy substrate under the conditions of Experiment 2.

In the initial stage, the solid-liquid interface emerged shortly after placing the water droplet was placed on the 6061 aluminum alloy surface, and moved rapidly from the bottom to the top of the droplet. During this process (images 1-7 of Fig. 6), no significant changes in contact area were observed, although the shape of the droplet did change. As the cooling continued, the height of the water droplet showed a marked increase and the liquid droplet froze, forming the solid ice (images 8-10 of Fig. 6). At the same time, the hemisphere shape of the droplet assumed a peculiar peach-like appearance. Frost started to form on the tip of the frozen droplet and gradually covered its entire outer-surface (images 11-15 of Fig. 6).
4. Discussion

In order to gain a better understanding of the ice adhesion, we applied the liquid-like layer (LLL) theory proposed by Michael Faraday (1859). The LLL separates the ice layer from the substrate, as shown in Fig. 7. This assumption has been validated by several studies (Engemann et al., 2004; Guerin et al., 2016; He et al., 2017; Mezger et al., 2008; Rosenberg, 2005).

![Image of Adhesion Model](image-url)

Fig. 7 Adhesion model based on the liquid layer theory.

We infer that different initial surface temperatures would cause a change in the properties of the LLL, which are influenced by the spreading ability of the droplet and the structural stability of ice.

4.1. Influence of contact area on ice adhesion strength

The comparison of the experimental conditions corresponding to Experiment 1 and Experiment 2 indicated that the aluminum alloy substrate in Experiment 1 had a higher surface energy, which resulted in a stronger interaction between surface and droplet. Therefore, the droplets spread more rapidly and resulted in a larger contact area in the case of Experiment 1. The LLL between ice and substrate would not freeze immediately,
enabling the water droplets to spread and extend the contact area (images 1-5 of Fig. 5). The capillary forces created by the LLL increased, entailing a higher energetic cost for the accreted ice on the substrate to be removed from the surface. On the other hand, the lower surface energy of the substrate in Experiment 2 resulted in a weaker interaction with droplets, and the frozen LLL would limit their spread on the Al-alloy surface (images 1-7 of Fig. 6).

4.2. Influence of contact stability on ice adhesion strength

In Experiment 1, the substrate and the droplets were simultaneously refrigerated, and the water droplets were in a supercooled state. Supercooled water does not freeze until the process is initiated by some external factors. Following the continuous reduction in the initial temperature of the surface, and the molecular energy of the water droplet would continuously decrease until the predetermined temperature was reached. Under the conditions of Experiment 1, the droplet spent a long time in the supercooled state. If the duration of the freezing process of the droplet on the cold surface was sufficiently long, the temperature difference between the two systems would change slowly, leading to a steady and prolonged heat exchange, and resulting in a more effective contact between the droplet and the surface; therefore, an enhanced stability of the frozen interface would be achieved under these conditions.

On the other hand in Experiment 2, the aluminum alloy substrate was cooled first. In this case, when a water droplet was rested on the cold surface, the latter would have a restraining effect on the liquid layer. In addition, the small size of the water droplet led
to low heat absorption, resulting in a shorter freezing time.

Furthermore, the temperature of the sample surface was much lower than the ambient temperature. After being dropped onto the surface, the droplet was rapidly cooled by the substrate surface, and its energy decreased. Due to the shorter freezing time, the temperature of the droplet changed rapidly, with a consequently shorter heat exchange time. Therefore, the water droplet froze before making sufficient contact with the Al alloy substrate. This rapid freezing process caused crack initiation, which in turn resulted in instability and deterioration of the contact interface between ice and substrate.

5. Conclusion

The ice adhesion strength of the water droplet on a cold substrate was measured by a purposely-built apparatus conform to the ASTM standard. We inspected the morphological changes of the droplet during the freezing process, and analyzed the influence of the initial surface temperature on the freezing characteristics of the droplets.

The analysis showed that the ice adhesion strength measured under the conditions of Experiment 1 was approximately twice that obtained in Experiment 2. This indicates that the ice adhesion strength decreased for a lower initial surface temperature under otherwise identical conditions, including final surface temperature and other test parameters, and forms a more stable contact between ice and the substrate in Experiment 1. Hence, it will need more energy or cost to remove the accumulated ice on
materials surface. Based on the experimental results and the inspection of the freezing process, the influence of the initial surface temperature on the ice adhesion strength can be attributed to two main causes: (1) the two test conditions involved a different effect of the cold surface on the water droplet during its freezing process, also with a different contact area between ice and substrate; (2) the large initial surface temperature difference of between substrate and droplet markedly affected the freezing time. In particular, a short freezing time resulted in unstable heat exchange between substrate and droplet, which in turn produced structural defects at the contact interface.

In conclusion, the experimental methods adopted in this paper simulate the influence of surface initial temperature on the freezing characteristics before and after using thermal de-icing. The results of the experiments demonstrate that surface initial temperature has a great influence on ice adhesion strength. The present paper provides an experimental basis for optimizing and reducing the cost of thermal de-icing processes, which will be highly useful within the rapid development of high-speed rail transport.

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