

Article

Experimental Study on the Adhesion Strength of the Frozen Ice for Aircraft Moving Parts

Xiaofeng Xue [†], Guoyan Qiang ^{*,†} , Yunwen Feng and Tiansu Luo

School of Aeronautics, Northwestern Polytechnical University, No. 127, Youyi West Road, Beilin District, Xi'an 710060, China

* Correspondence: 13920597137@163.com

[†] These authors contributed equally to this work.

Abstract: At alpine regional airports, aircraft are covered with frozen ice when they encounter extreme weather such as heavy snow or frost. The movement parts of aircraft cabin doors, flaps and landing gear may be affected due to the infiltration of freezing ice, and the movement stagnation may occur when the the accumulation of ice is more serious. This paper sets up a mechanical performance test of frozen ice for this engineering problem to provide data that is beneficial to the selection of the mechanism drive and the determination of ice-breaking loads. The test is conducted based on the standard tensile shear test. In order to overcome problems such as the poor icing effect of the traditional specimen or the easy damage of the specimen ice, we improved the structure of the specimen and the method of the test. According to the characteristics of growth of frozen ice, we introduced freezing time, type of water quality and adhesion materials as test variables. The results show that: the ice adhesion strength of frozen ice increases and then decreases ($-15^{\circ}\text{C}\sim-55^{\circ}\text{C}$). At the ambient temperature of $-15^{\circ}\text{C}\sim-55^{\circ}\text{C}$ and freezing for 2 h~6 h, the ice adhesion strength of aluminum alloy surface ranges from 0.009 MPa to 0.568 MPa, and that of frozen ice on a silicone rubber surface is 0.005 MPa~0.147 MPa. The duration of freezing did not significantly affect the adhesion strength of frozen ice. Among the three water qualities, the frozen ice from distilled water has the greatest adhesion strength, the lake water is the most medium, and the sea water is the smallest. The results of this test can be widely used in the determination of the ice-breaking load of civil aircraft, amphibious aircraft, ships, and the design of anti-ice/de-icing systems.

Keywords: ice adhesion strength; frozen ice; ambient temperature; duration of freezing; type of water quality; aviation materials



Citation: Xue, X.; Qiang, G.; Feng, Y.; Luo, T. Experimental Study on the Adhesion Strength of the Frozen Ice for Aircraft Moving Parts. *Aerospace* **2022**, *9*, 589. <https://doi.org/10.3390/aerospace9100589>

Academic Editor: Ning Zhao

Received: 24 August 2022

Accepted: 21 September 2022

Published: 11 October 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Parking at airports in northern Heilongjiang province, the Qinghai-Tibet Plateau and Xinjiang province, aircraft such as the ARJ21-700, which is well-selling in the Chinese mainland, and the AG-600, for forest fire fighting and maritime rescue, will not take off normally, and the surface of the aircraft could be covered with frozen ice for hours of exposure in extreme weather such as heavy snow, frost drop and freezing rain. When the ice on the surface of aircraft increases to a certain extent, it may cause the failure of opening cabin doors, wings, flaps, seams and even engine blades can be frozen. In addition, amphibious aircraft and ships sailing at high latitudes also face a heavy amount of work over de-icing or ice-breaking for a long time.

Researchers have studied the influence of surface roughness of the adhesion surfaces, environmental temperature, ice type, ice density and other factors on the ice adhesion strength, and found that: the ice adhesion strength increases with the surface roughness; the ice adhesion strength does not show a linear relationship with ambient temperature, the liquid water content, the annealing time and other variables, the ice adhesion strength increases flat with the section speed; and the ice adhesion strength decreases with the increase

of the water contact angle [1–3]. Gagnon, Robert et al. [4] carried out ice adhesion tests that were conducted on five substrates with differing ice-phobic coatings. Rectangular-plate ice samples, freeze-bonded onto the surfaces, were pushed from one edge at 0.5 mm/s until shear-detachment occurred. Test results at $-12\text{ }^{\circ}\text{C}$ showed a wide variation of ice adhesive strength between the coatings (0.022~0.216 MPa). At $-22\text{ }^{\circ}\text{C}$, the preparation method usually led to the contact region of ice samples that were only partially bonded to the outer surface of the ice-phobic coatings of the substrates. For saline ice generated by spraying at $-12\text{ }^{\circ}\text{C}$ and $-22\text{ }^{\circ}\text{C}$, no freeze-bonding occurred on any of the surfaces. Rnneberg, Sigrid et al. [5] investigated the effect of different types of ice on ice adhesion strength. The ice adhesion strength is measured with a centrifugal adhesion test and varied from $0.78 \pm 0.10\text{ MPa}$ for precipitation ice, $0.53 \pm 0.12\text{ MPa}$ for in-cloud ice to $0.28 \pm 0.08\text{ MPa}$ for bulk water ice. Additionally, the results indicate that the ice adhesion strength inversely correlates with the density of ice. Rebekah G. Douglass et al. [6] found that a higher impact velocity and higher surface roughness would lead to a higher adhesion strength of impact ice on isotropic metals. M.L.A. Pervier et al. [7] proposed a new shear test for ice environment, forming ice on the fixture containing the sample material, and then forced loading and damage. Taking the maximum test value, it was found that the adhesion shear strength value of alloy impact on ice with a different surface finish was between 2~14 MPa. Matsushita, Hisao [8] performed the adhesion force and shear strength measurements on the sea ice, with an adhesion strength of around 50 kPa and a shear strength of between 0.2 and 0.3 MPa. M.C. Chu et al. [9] found that the adhesion strength of frost and glaze ice was 0.12~0.41 MPa, a weak statistical linear correlation between wind speed and droplet size, and the shear strength of the ice was independent of the air temperature of the tunnel, the thickness of the accumulation, and the material of the substrate. Chen, Tingkun et al. [10] found that the ice bonding strength at aluminum alloy surfaces at room temperature was twice that at cooler surfaces (e.g., $5\text{ }^{\circ}\text{C}$), because surface water droplets at a higher initial temperature can be diffuse enough and can form a large contact area. Zhang, Y.J. [11] found that the ice shear strength of fresh water first increases and then decreases as the ambient temperature decreases; the ice adhesion shear strength of seawater decreases linearly as the temperature decreases. Jin Jingfu et al. [12] found that the elastic modulus, elasticity coefficient, and distribution of the coating all significantly affected the ice adhesion strength on the substrate. The icing adhesion strength can be reduced by increasing the surface elasticity of the material.

Furthermore, Dawood, Bishoy et al. [13] established a test framework using single cantilever, straight shear and push shear tests to study the effect of test methods on ice adhesion strength, and found that the apparent toughness of the zero-angle push-in test was an order of magnitude higher than the straight shear test. Beeram, Prashanth et al. [14] used the ice shear test mechanism to measure the ice adhesion strength, which studied the effectiveness of different surface and superhydrophobic coatings, and verified the ice adhesion test model. Pan Huan [15] used neural network technology to build an ice-type prediction model. Zhang Yongjie [16] has summarized the common methods used to measure the mechanical properties of ice. Dong, Yiqun [17] applies a deep neural network to flight parameter identification to detect and characterize aircraft icing conditions. De-icing technology is expensive and time-consuming. In order to reduce the cost, time and physical labor related with deicing, researchers have conducted a lot of research on the ice adhesion strength [18], but most of the experiments have focused on the impact ice in flight. The ice type and consideration factors of the impact ice do not apply to the frozen ice, and the lack of factors such as extreme environmental temperature, freezing time, water quality type, and adhesive material are considered. In view of the above engineering problems, this paper sets up the frozen ice mechanical performance test, and the test data can provide data support for the drive type selection and ice-breaking load determination of the typical aircraft mechanism.

2. Test Design of the Adhesion Strength of the Frozen Ice

2.1. Experiment Design

The nature of this test is a component-level static test, and the basic information of the test is shown in Table 1.

Table 1. Basic information of test.

Number	Project	Content
1	Test objective	Measure the shear adhesion strength of the aviation material to the ice layer
2	Trial basis	Outline of Mechanical Property of Civil Machine; GB/T 13936-2014 Methods for Determination of Tensile Shear Strength of Rubber Rubber and Metal
3	Test item	The ice adhesion strength test of aluminum alloy-ice-aluminum alloy (AL-Ice-AL); The ice adhesion strength test of aluminum alloy-ice-silicone rubber (AL-Ice-SR)
4	Laboratory environment	Temperature: 23 °C ± 3 °C; Humidity: 50% RH ± 10% RH
5	Proving time	December 2021–March 2022

To calibrate the ice-breaking load at the separation surface of cabin doors, landing gear and moving wing surface, the test extracts the materials of moving parts separation surface (such as aluminum alloy, silicone rubber of sealing belt) to make test parts, studying the adhesion strength of frozen ice to different materials under different environmental temperatures, freezing times, and types of water quality.

Given that the ground atmospheric environment may contain impurities and the water quality of amphibious aircraft is more complex, this test used three kinds of water quality (distilled water, lake water and seawater), where the lake water is the surface water after the rain moat used to simulate the frozen ice of the aircraft and the frozen ice of the amphibious aircraft berthing. To verify the statement that “the longer the freezing time, the stronger the freezing time is”, the duration of freezing is introduced as the test variable. In addition, the test combines the ambient temperature of the civilian machine to freeze the ice. For the parameters such as water droplet diameter, surface roughness of material, loading rate, type of ice, etc., affecting the mechanical properties of frozen ice but not included in the test variable study table, we assigned them fixed values according to the actual working conditions of the aircraft.

At present, there are four universal test methods for the mechanical properties of the ice block, namely: straight pull shear, direct push shear, cylindrical push shear and cylindrical pull shear. Among them, the cylindrical push shear and the cylindrical pull shear are beneficial to the formation of uniform impact ice, so it is mostly used to study the impact ice, and the cost is high. The straight pull shear has the lowest cost and convenient operation, which is also the choice of this test.

The test matrix elements include test items, test equipment, test implementation method and test operation process. Among them, the test items are divided into two categories according to the substrate materials of the test items, namely, aluminum alloy–ice–aluminum alloy static test (test of AL-Ice-AL) and aluminum alloy–ice–silicone rubber static test (test of AL-Ice-SR). The preparation, icing, insulation and tensile shear failure of specimens are conducted according to the test items. The test piece information is shown in Table 2, and the test breakdown item matrix is shown in Table 3.

Table 2. Test piece information sheet.

Project	AL-Ice-AL Static Test Piece	AL-Ice-SR Static Test Piece
Sample composition	Two aluminum alloy plates	An aluminum alloy plate and an silicone rubber board
Material of test piece	7050-T7451	7050-T7451/TX-FROL 50
Size of test piece	25 × 25 × 10	25 × 25 × 10
Use of test piece	Test the ice adhesion strength on the aluminum alloy surface	Test the ice adhesion strength on the silicone rubber surface

Table 3. Test the subdivision item matrix table.

Test Item	Sub-Items of the Experiment	Freeze for 2 h	Freeze for 4 h	Freeze for 6 h
Static test of AL-Ice-AL	Distilled water at −15 °C	2	2	2
	Distilled water at −25 °C	3	3	3
	Lake water at −25 °C	3	3	3
	Sea water at −25 °C	3	3	3
	Distilled water at −35 °C	3	3	3
	Distilled water at −45 °C	3	3	3
	Distilled water at −55 °C	2	2	2
Static test of AL-Ice-SR	Distilled water at −15 °C	2	2	2
	Distilled water at −25 °C	3	3	3
	Lake water at −25 °C	3	3	3
	Sea water at −25 °C	3	3	3
	Distilled water at −35 °C	3	3	3
	Distilled water at −45 °C	3	3	3
	Distilled water at −55 °C	2	2	2

Note: The lake water is taken from the Xi'an moat river, and the sea water is taken from Bohai Bay, China.

2.2. Test Equipment

The equipment and instruments selected in the test are all calibrated and measured within the validity period. The test equipment information is shown in Table 4.

Table 4. Test equipment and test instruments.

Number	Device	Unit Type	Use
1	Temperature and humidity meter	HTC-1	Measure ambient humidity
2	Number of vernier calipers	0~150 mm	Measure the ice specification
3	Damand heat test box for high and low temperature alternating	GDJS-1000	Make frozen ice specimens
4	Electronic universal test machine	UTM5205HB	Apply the load
5	Type S sensor	BSS-200 kg	Measure the ice-breaking load
6	Test machine environment box	TS-160	Provide the test temperature environment for the specimen
7	Camera	-	Take a picture

2.3. Implementation Method of the Test

The implementation method specifically includes two parts: specimen design and production, specimen icing and thermal insulation.

2.3.1. Test Piece Design and Production Principle

Because of the size effect of the ice itself, the larger the size, the lower the strength of the ice [19], and the test uses the standard piece size of GB/T 13936-2014 test. The specification of the test ice layer is $25 \times 25 \times 2 \text{ mm}^3$.

The static test piece of each AL-Ice-AL consists of two pieces of aluminum alloy and a piece of ice. The test piece requires a uniform roughness ($R_a = 1.6$) on the icing surface. The Specimens in the experiment were formed in batches by a precision milling process, and the roughness of each specimen was measured successively by TR200 roughness tester (indicating value error $\pm 10\%$, indicating value variability $\pm 6\%$). Three points on the adhesion surface of a specimen were randomly selected. If the mean roughness of the three points was less than $R_a = 1.6$, the test piece was considered to be qualified. The specimen is used to simulate the frozen ice adhesion conditions on the surface of the aircraft motion mechanism. Additionally, the icing effect of the specimen in the beginning of the test shows that the greater the mass of the metal block, the easier it is to adhere to the ice layer on the specimen surface, and the stronger the ice layer adhesion relationship is. The test is to let the test block reduce to the predetermined freezing temperature, and then through the drop method between the metal or between the metal block and the silicone rubber block to form ice in the process of water freezing, which will release a certain amount of heat. At this point, the most direct and rapid way of heat dissipation is to the attached metal block, while the heat dissipation through the environment is relatively slow. Therefore, on the one hand, under the condition of certain thermal conductivity and other factors, the larger the mass of the metal block, the easier it is to absorb the heat released by water freezing, and the more beneficial it is to icing. On the other hand, the increase of the thickness of the test block will also lead to the additional bending moment in the tensile shear test. When the additional bending moment increases to a certain extent, the tangential failure test may degenerate into a combined tangential and normal failure test, and the obtained shear adhesion strength will no longer be reliable. After repeated attempts, we found that the test piece under 10 mm thickness has a good ice effect relatively.

The static test piece of each AL-Ice-SR consists of a piece of aluminum alloy, a piece of silicone rubber and a piece of ice. The test silicone rubber blocks are processed and manufactured in accordance with the HG6-677-74 national defense industry silicone rubber standard. The machine factory we are working with has a TR200 roughness detector and other monitoring equipment to ensure that the surface quality of the test parts reaches the level of the door sealing belt. The specimen is used to simulate the door with sealed seal attached by frozen ice. Considering the relatively small stiffness of the silicone rubber, a large deformation may occur in the test process, and the deformation will produce an uneven stress in the frozen area and lead to the error of the shear test. Therefore, a raised structure is designed on the upper and lower non-adhesion surfaces of the silicone rubber block, and the structure is combined with the fixture, so as to prevent the large lateral sliding of the silicone rubber block in the test process and ensure the test accuracy.

2.3.2. Method of Specimen Icing and Thermal Insulation

The ambient temperature of the outer field is combined by many factors such as sunshine and atmospheric conditions, and the ambient temperature makes nonlinear complex changes with time. Therefore, the simulation process of temperature change in freezing environment is more complicated.

Janjua, Zaid A. [20], University of Nottingham, studied the effect of ambient temperature and freezing temperature on adhesion strength, and found that the ambient temperature was much greater than the effect of freezing temperature on icing adhesion strength. Therefore, the secondary factor that is named the ice-making temperature can be simplified as a fixed value that is set above the test temperature point ($10^\circ\text{C} \pm 3^\circ\text{C}$), and the cooling rate shall be less than or equal to 3°C per minute. The experiment simulated the formation process of freezing ice by a dropping method. When the mold is filled with the frozen ice layer, move the specimen to the specified temperature point environment

($-55\text{ }^{\circ}\text{C}$, $-45\text{ }^{\circ}\text{C}$, $-35\text{ }^{\circ}\text{C}$, $-35\text{ }^{\circ}\text{C}$, $-25\text{ }^{\circ}\text{C}$, $-15\text{ }^{\circ}\text{C}$) for the specified duration (2 h, 4 h, 6 h). Frozen ice specimens are shown in Figure 1.

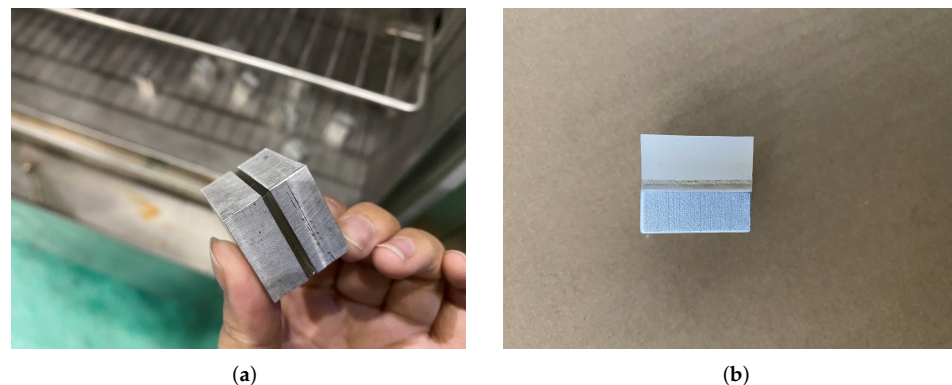


Figure 1. Ice diagram of specimens. (a) AL-Ice-AL specimen. (b) AL-Ice-SR specimen.

2.4. Process of the Test Operation

The test steps are as follows:

(1) Put the test piece into the icing mold, add the leakage-proof sealing glue in the predetermined icing area, and then put it into the test box with high and low temperature alternating humidity, and make the ice layer grow in the shear test area of the specimen through the drop point ice method;

(2) Measure the actual ice thickness, width and length value of the ice test piece in the incubator, which should be accurate to 0.1 mm;

(3) According to the information of the test items in Table 3, the specimens are kept warm for the corresponding time and at the corresponding ambient temperature. The temperature values of the test box and test fixture are also adjusted to the test temperature of the specimen ($-15\text{ }^{\circ}\text{C}$, $-25\text{ }^{\circ}\text{C}$, $-35\text{ }^{\circ}\text{C}$, $-45\text{ }^{\circ}\text{C}$, $-55\text{ }^{\circ}\text{C}$), and the test box will be kept warm for half an hour after reaching the predetermined temperature;

(4) Install the specimen quickly on the fixture in the test box. Xiao, Z. [21] found that the tensile strength of ice samples would basically not change. However, when the strain rate exceeds 0.01/s and the brittle property of ice is still maintained, the tensile strength of ice decreases with the increase of strain rate. Considering the experimental efficiency and the possibility of obtaining a larger experimental value, the strain rate was chosen as 0.01/s in this experiment. By multiplying the strain rate by the length of the specimen and rounding the result, the tensile speed of the tensile machine is 0.3 mm/s (18 mm/min), so the tensile machine needs to adjust the load at a rate of 18 mm/min until a large displacement of more than 5 mm between the ice and the test piece, which can obtain a relatively large ice adhesion strength value of the specimen. Figure 2a showed the effect of the icing specimen mounted on the fixture and tension machine;

(5) Record the load curve of the S-type force sensor, observe the ice failure position of the shear specimen, and record it;

(6) Return to the test machine and fixture, repeat steps 1~5 to conduct the adhesion strength test of the frozen ice in the next test piece until all the test pieces are completed.

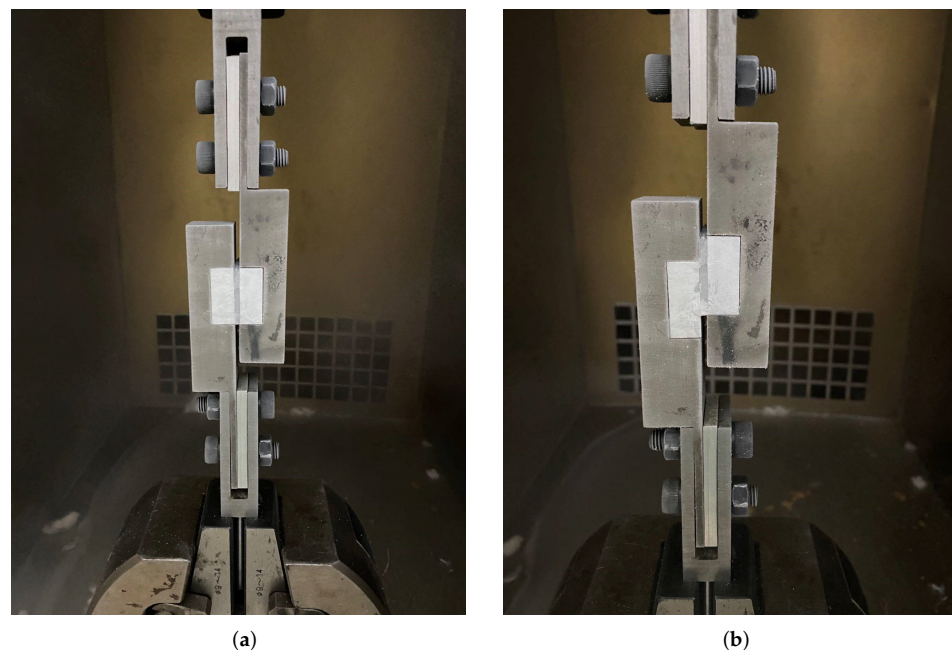


Figure 2. Assembly and loading drawing of test parts and fixture. (a) Drawing of test parts and fixtures. (b) Effect diagram of the tensile load destroying the adhesive ice layer.

3. Key Technique for the Adhesion Strength Test of Frozen Ice

The test of the shear adhesion strength of frozen ice is different from the test of the shear adhesion strength of conventional materials. On the one hand, the ice layer is very fragile and easily damaged, which brings many problems to the specimen ice making, specimen handling and specimen card loading; on the other hand, the artificial freezing into uniform ice on the aviation material surface (aluminum alloy and silicone rubber) is difficult, and the ice structure is easily damaged during the mold removal stage. This test has formed the key technique in overcoming the above-mentioned difficulties. That is: the test technology of the adhesion strength of frozen ice on metal or non-metal.

In view of the experimental difficulties, such as forming ice on the surface of the aircraft moving parts or the poor effect of ice, we have improved and designed the new test parts, and the installation effect of the new test parts is shown in Figure 2a.

The structure of test piece is divided into the icing module and the support module, and it is processed and installed separately. The contact installation method when pressed in from the front side can effectively avoid the impact force during in the installation process of the support module, and also greatly shorten the test time. The fixture is installed in the test low temperature box in advance, the test piece is pressed into the cavity, the tensile machine slowly breaks the ice and measures the ice-breaking load, then we throw out the measured test piece, reset the fixture, complete a test, and wait for the next test piece to press in. The design of loading the test piece abandons the repeated loading and dismantling operation of the traditional fixture, which greatly accelerates the test speed and avoids the damage of the ice layer during the installation of the fixture. In addition, turning the icing module of the test piece into a compact cube structure is very beneficial for the ice making, mold unloading and handling of the specimen.

4. Results and Analysis of the Adhesion Strength Test of Frozen Ice

The ice damage form of the adhesion surface of aviation material and the load value of the ice-breaking can be tested. The adhesion strength of the frozen ice is obtained by substituting the load of the ice-breaking and the shear surface size of the ice into the physical formula. Analysis of variance (ANOVA) is used to investigate whether the effect of test variables on the adhesion strength of frozen ice is significant, combining the adhesion

strength of the frozen ice with the geometry of the ice shape of the separation surface of the aircraft moving parts.

4.1. The Result of the Shear Destruction of Frozen Ice

4.1.1. Shear-Section Situation of the Ice Layer

The section of the frozen ice of aviation materials is shown in Figures 3 and 4.

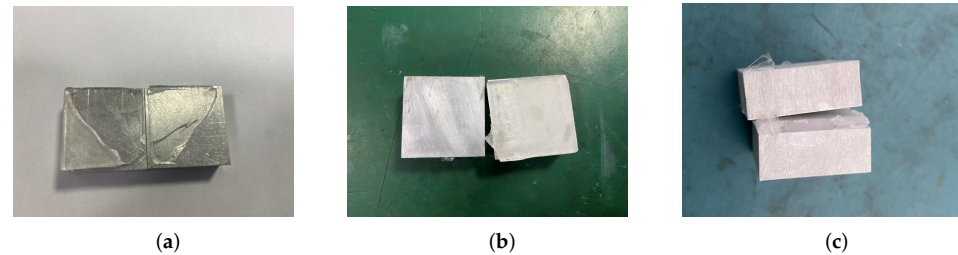


Figure 3. Section view of tensile shear disruption of AL-Ice-AL specimens. (a) Experimental section of specimen frozen for 6 h at -15°C . (b) Experimental section of specimen frozen for 2 h at -25°C . (c) Experimental section of specimen frozen for 2 h at -55°C .

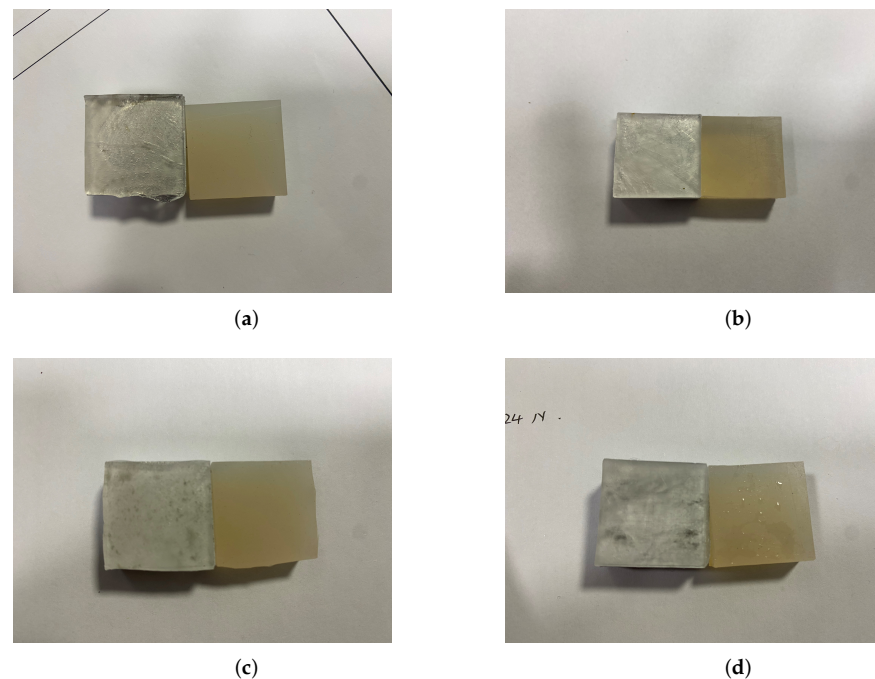


Figure 4. Section view of tensile shear disruption of AL-Ice-SR specimens. (a) Experimental section of specimen frozen (by distilled water) for 2 h at -55°C . (b) Experimental section of specimen frozen by distilled water for 2 h at -25°C . (c) Experimental section of specimen frozen by lake water for 2 h at -25°C . (d) Experimental section of specimen frozen by sea water for 2 h at -25°C .

Internal shear disruption inside the ice is shown in Figure 3a. However, its section is not neat enough, so it cannot be ruled out that it is a mixed failure form composed of cohesive failure and adhesive failure. Therefore, the value of icing adhesion strength and ice shear strength at this time may be similar or comparable. However, Figure 3b shows that the ice adhesion surface falls. It can be seen that the adhesion strength of frozen ice at -15°C is similar or comparable to the shear strength, and that of frozen ice at -25°C is less than the shear strength.

4.1.2. Ice-Breaking Load of Frozen Ice of Aviation Material

The ice-breaking load is measured by the electronic universal test machine (GT-S-17). The average and peak data have been extracted for the values under the single test conditions. The specific test readings of the two types of trials are shown in Tables 5 and 6.

Table 5. Ice-breaking load of AL-Ice-AL specimens in shear tensile test.

Ambient Temperature (°C)	Freeze Time (h)	The Form of Destruction	Average Value of the Ice-Breaking Load (N)	Maximum Value of the Ice-Breaking Load (N)	Frozen Area (mm ²)
−15	2	Shear destruction inside the ice layer	235.0	267.0	620.38
	4	Shear destruction inside the ice layer	124.0	138.0	624.38
	6	Shear destruction inside the ice layer	177.1	222.0	620.01
−25	2	The adhesion surface falls off	110.2	150.0	622.25
	4	The adhesion surface falls off	189.1	369.1	620.92
	6	The adhesion surface falls off	352.3	417.0	620.76
−25 (lake water)	2	The adhesion surface falls off	105.9	150.0	622.50
−25 (sea water)	2	The adhesion surface falls off	5.7	6.1	621.75
	2	The adhesion surface falls off	82.0	110.0	622.25
	4	The adhesion surface falls off	30.7	62.8	620.92
−35	6	The adhesion surface falls off	35.9	42.1	620.76
	2	The adhesion surface falls off	46.6	89.2	622.25
	4	The adhesion surface falls off	28.7	39.7	620.92
−45	6	The adhesion surface falls off	21.2	23.6	620.76
	2	The adhesion surface falls off	5.8	6.0	620.38
	4	The adhesion surface falls off	37.4	38.4	624.38
−55	6	The adhesion surface falls off	16.7	19.7	620.01

Table 6. Ice-breaking load of AL-Ice-SR specimens in shear tensile test.

Ambient Temperature (°C)	Freeze Time (h)	The Form of Destruction	Average Value of the Ice-Breaking Load (N)	Maximum Value of the Ice-Breaking Load (N)	Frozen Area (mm ²)
−15	2	The adhesion surface falls off	18.7	19.6	623.13
	4	The adhesion surface falls off	18.1	20.6	623.50
	6	The adhesion surface falls off	53.4	54.3	624.62
−25	2	The adhesion surface falls off	88.2	99.8	623.25
	4	The adhesion surface falls off	51.3	78.1	624.25
	6	The adhesion surface falls off	91.6	103.0	624.67
−25 (lake water)	2	The adhesion surface falls off	35.3	53.4	624.75
−25 (sea water)	2	The adhesion surface falls off	3.2	4.2	624.75
	2	The adhesion surface falls off	32.1	45.0	623.25
	4	The adhesion surface falls off	46.3	72.2	624.25
−35	6	The adhesion surface falls off	62.4	68.9	624.67
	2	The adhesion surface falls off	51.8	80.0	623.25
	4	The adhesion surface falls off	56.5	63.5	624.25
−45	6	The adhesion surface falls off	41.4	44.5	624.67
	2	The adhesion surface falls off	36.2	41.9	623.13
	4	The adhesion surface falls off	37.4	38.4	624.38
−55	2	The adhesion surface falls off	16.7	19.7	620.01

Note: AL-Ice-SR specimens all fall off of the ice layer on the adhesion surface of silicone rubber. The adhesion strength of −55 °C is so small that the mold cannot be removed, so the ice-breaking load is 0.

4.2. Analysis of the Shear Adhesion Strength of Frozen Ice

This test measured the ice adhesion strength by direct shear test. The tensile shear adhesion strength of AL-Ice-AL or AL-Ice-SR is calculated as the formula:

$$\tau = \frac{p}{L \times w} \quad (1)$$

where: τ is adhesion strength in unit of MPa; p is the ice-breaking load of the sample in units of Newton; L is the length of the sample adhesion surface in mm; and w is the width of the adhesive surface of the sample in mm.

Note: The sample has two adhesive surfaces. The tensile shear strength was calculated from the size of the frozen ice on one adhesive surface where the separation actually occurred.

From the test data in Tables 5 and 6 to Equation (1), the adhesion strength value of the frozen ice under different working conditions is shown in Tables 7 and 8, and the trend of the adhesion strength of the frozen ice is shown from Figures 5–7.

As can be seen from Table 5, the ice failure form under $-15\text{ }^{\circ}\text{C} \sim -25\text{ }^{\circ}\text{C}$ changes from internal shear failure to adhesion surface shedding, that is, when the ambient temperature is between $-15\text{ }^{\circ}\text{C} \sim -25\text{ }^{\circ}\text{C}$, the adhesion strength of the aluminum alloy specimen with surface icing is equivalent to the shear strength of the ice layer itself. When the temperature is below $-25\text{ }^{\circ}\text{C}$, the specimen adhesion strength is less than the ice shear strength. According to Table 6, the shear strength of $-15\text{ }^{\circ}\text{C} \sim -55\text{ }^{\circ}\text{C}$ ice layer is greater than the ice adhesion strength of silicone rubber specimens.

Compared with Table 5 and Table 6, we can see that the ice adhesion strength of the aluminum alloy surface is much greater than that of the silicone rubber surface, so the ice layer falls off from the side of the silicone rubber adhesion surface in Table 6. In addition, the surface of $-55\text{ }^{\circ}\text{C}$ silicone rubber was found to be difficult to freeze ice.

Table 7. Ice-breaking load of AL-Ice-AL specimens in shear tensile test.

Ambient Temperature ($^{\circ}\text{C}$)	Freeze Time (h)	Mean Adhesion Strength of Frozen Ice (MPa)	Maximum Value of Adhesion Strength for Frozen Ice (MPa)
−15	2	0.379	0.477
	4	0.199	0.221
	6	0.285	0.358
−25	2	0.177	0.243
	4	0.305	0.594
	6	0.568	0.672
−25 (lake water)	2	0.170	0.241
−25 (sea water)	2	0.009	0.011
−35	2	0.132	0.177
	4	0.049	0.101
	6	0.058	0.068
−45	2	0.075	0.143
	4	0.046	0.064
	6	0.034	0.038
−55	2	0.009	0.010
	4	0.060	0.062
	2	0.027	0.032

Note: The mean value here refers to the mean value of frozen ice adhesion strength measured by repeated tests.

Table 8. Ice-breaking load of AL-Ice-SR specimens in shear tensile test.

Ambient Temperature (°C)	Freeze Time (h)	Mean Adhesion Strength of Frozen Ice (MPa)	Maximum Value of Adhesion Strength for Frozen Ice (MPa)
−15	2	0.030	0.031
	4	0.029	0.033
	6	0.085	0.087
−25	2	0.142	0.160
	4	0.082	0.125
	6	0.147	0.165
−25 (lake water)	2	0.057	0.085
−25 (sea water)	2	0.005	0.007
−35	2	0.051	0.072
	4	0.074	0.116
	6	0.100	0.110
−45	2	0.083	0.128
	4	0.091	0.102
	6	0.066	0.071
−50	2	0.058	0.067
	4	0.085	0.103
−55	2	0.009	0.010

As can be seen from Figure 5, as the ambient temperature drops ($-15^{\circ}\text{C} \sim -55^{\circ}\text{C}$), the freezing adhesion strength of aluminum alloy first increases and then decreases, and the peak is about -25°C . The results are consistent with the results of the ice adhesion strength test designed by Ding Liang [22] ($0.051\text{ MPa} \sim 0.255\text{ MPa}$ at -13°C) and the test trend of Zhang, Y.J. [16] that the shear strength of fresh water first increases and then decreases with the ambient temperature decrease. Therefore, the test data are relatively reliable. In this test, the frozen ice was artificially generated by the dripping method, and the shear adhesion strength of the ice was measured by the tensile shear method. The high and low temperature alternating damp-heat test chamber was used to create environmental conditions for the specimens, and the tensile failure was carried out by the electronic universal testing machine. The experimental results show that the mean adhesion strength of the frozen ice ranges from 0.009 MPa to 0.568 MPa when the ambient temperature is -55°C to -15°C for 2 h to 6 h and the adhesion surface roughness of aluminum alloy is up to 1.6 microns, and the maximum value of ice adhesion strength test ranges from 0.010 MPa to 0.672 MPa .

As can be seen from Figure 6, the icing adhesion strength on the silicone rubber surface with the ambient temperature decreasing ($-15^{\circ}\text{C} \sim -55^{\circ}\text{C}$) under the same freezing time first increases and then decreases, reaching a peak at around -25°C . The average test value of silicone rubber icing adhesion strength is between 0.005 MPa and 0.147 MPa , and the maximum test range is $0.007\text{ MPa} \sim 0.165\text{ MPa}$.

As can be seen from Figure 7, the icing adhesion strength of aluminum alloy or silicone rubber is affected by water quality. It has the largest icing adhesion strength of distilled water, followed by lake water and the smallest sea water. At the same temperature, the adhesion strength of lake ice is 78.41% of distilled water ice and seawater ice is 4.55% of distilled water ice. The adsorption of impurity or salt on the grain surface on frozen ice nucleation may cause such test phenomenon.

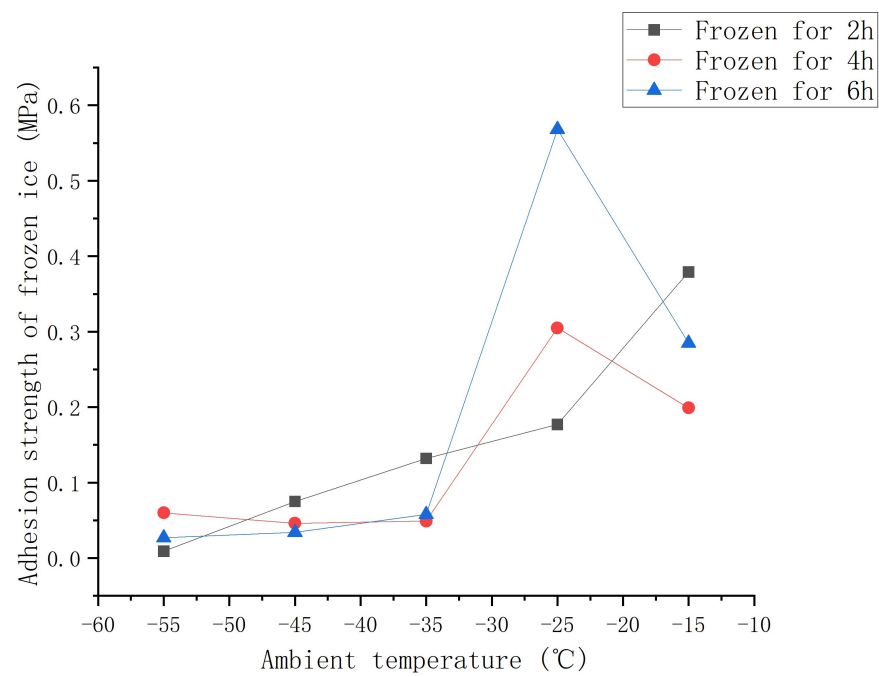


Figure 5. Adhesion strength of frozen ice on the aluminum alloy surface.

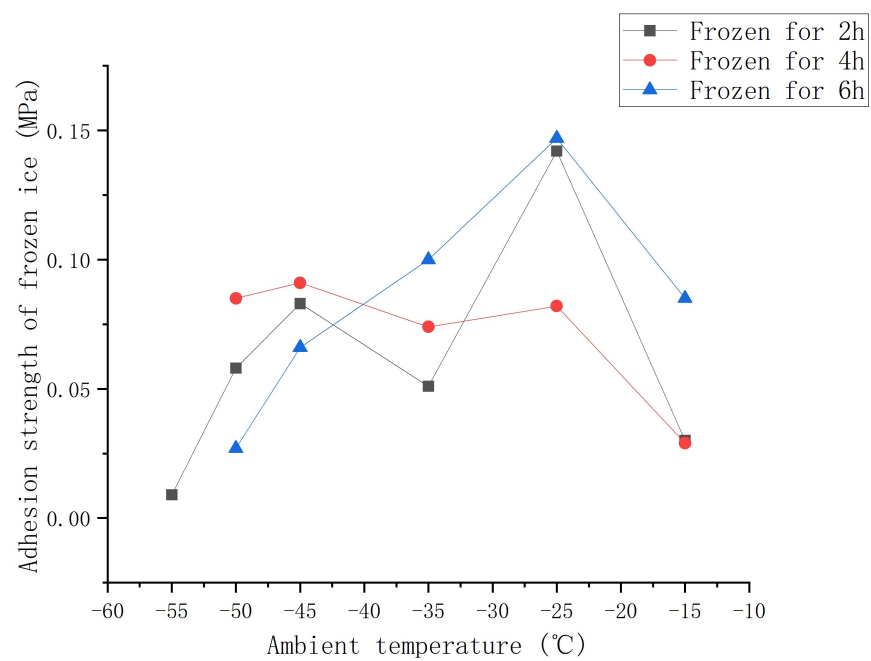


Figure 6. Adhesion strength of frozen ice on the silicone rubber surface.

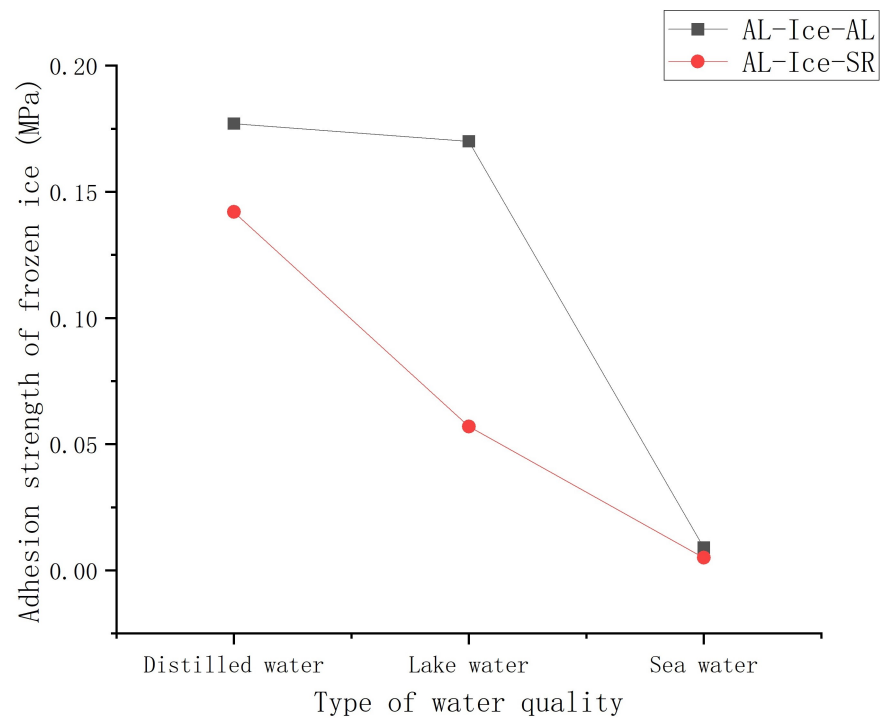


Figure 7. Adhesion strength of frozen ice under different water quality and different materials.

4.3. Significance Analysis of the Influencing Factors

Let the influence factor A be the ambient temperature, with r different levels of $A_1, A_2, \dots, A_r$. Factor B is the freezing duration, with s different levels of $B_1, B_2, \dots, B_s$. Three trials were repeated at each combination level AB, measuring the test data as X_{ij} . The results are shown in Tables 9 and 10; let the influence factor C be the type water quality, with 3 different levels of C_1, C_2, C_3 . Factor D is the type of material being adhered to, with two different levels of D_1, D_2 . Three trials were repeated at each combination level CD, measuring the test data as Y_{ij} . The results are shown in Table 11.

Table 9. Adhesion strength of AL-Ice-AL test piece.

Project	Freezing 2 h (B_1)	Freezing 4 h (B_2)	Freezing 6 h (B_3)
−25 °C (A_1)	0.175, 0.243, 0.113	0.148, 0.592, 0.172	0.592, 0.439, 0.639
−35 °C (A_2)	0.122, 0.177, 0.096	0.017, 0.030, 0.102	0.057, 0.049, 0.068
−45 °C (A_3)	0.143, 0.029, 0.052	0.064, 0.047, 0.028	0.032, 0.038, 0.032

Table 10. Adhesion strength of AL-Ice-SR test piece.

Project	Freezing 2 h (B_1)	Freezing 4 h (B_2)	Freezing 6 h (B_3)
−25 °C (A_1)	0.135, 0.130, 0.160	0.011, 0.125, 0.111	0.116, 0.164, 0.159
−35 °C (A_2)	0.039, 0.043, 0.072	0.042, 0.116, 0.065	0.110, 0.090, 0.099
−45 °C (A_3)	0.057, 0.064, 0.128	0.077, 0.102, 0.093	0.007, 0.071, 0.057

Table 11. Test data of the orthogonal relationship between water quality and material.

Project	AL-Ice-AL (D_1)	AL-Ice-SR (D_2)
Distilled water (C_1)	0.175, 0.243, 0.113	0.135, 0.130, 0.160
Lake water (C_2)	0.243, 0.100, 0.168	0.529, 0.031, 0.085
Sea water (C_3)	0.009, 0.008, 0.011	0.007, 0.005, 0.003

We used Equations (2) and (3) to analyze the test data from Tables 9–11, and the analysis results are shown in Tables 12–14.

$$\left\{ \begin{array}{l} T = \sum_{i=1}^r \sum_{j=1}^s \sum_{k=1}^t X_{ijk} \\ P = \frac{1}{rst} T^2 \\ U = \frac{1}{st} \sum_{i=1}^r \left(\sum_{j=1}^s \sum_{k=1}^t X_{ijk} \right)^2 \\ V = \frac{1}{rt} \sum_{j=1}^s \left(\sum_{i=1}^r \sum_{k=1}^t X_{ijk} \right)^2 \\ R = \frac{1}{t} \sum_{i=1}^r \sum_{j=1}^s \left(\sum_{k=1}^t X_{ijk} \right)^2 \\ W = \sum_{i=1}^r \sum_{j=1}^s \sum_{k=1}^t (X_{ijk})^2 \end{array} \right. \quad (2)$$

where: r and s , respectively, represent the number of levels corresponding to the two factors in ANOVA, and t represents the number of repeated trials.

$$Q_A = U - P, Q_B = V - P, Q_{A \times B} = R - U - V + P, Q_E = W - R, Q_T = W - P \quad (3)$$

where: Q_A is the sum of squares between groups for factor A, Q_B is the sum of squares between groups for factor B, and $Q_{A \times B}$ is the sum of distance squares under the interaction between factor A and factor B.

Table 12. Analysis of variance of adhesion strength test of frozen ice on aluminum alloy surface.

Source of Variance	The Sum Of Squares of Deviations	Degree of Freedom	Mean of the Sum of Squares of Deviations	The Value of F	Significance Level
Factor A	0.4873	2	0.2437	24.71	Yes
Factor B	0.0480	2	0.0240	2.43	No
Interactions of A and B	0.2050	4	0.0513	5.20	Yes
Error	0.1775	18	0.0099		
Total sum value	0.9178	26			

Table 13. Analysis of variance of adhesion strength test of frozen ice on silicone rubber surface.

Source of Variance	The Sum of Squares of Deviations	Degree of Freedom	Mean of the Sum of Squares of Deviations	The Value of F	Significance Level
Factor A	0.0127	2	0.0064	6.79	Yes
Factor B	0.0021	2	0.0011	1.12	No
Interactions of A and B	0.0100	4	0.0025	2.66	No
Error	0.0169	18	0.0009		
Total sum value	0.0417	26			

Table 14. Analysis of variance for orthogonal tests of different water quality and materials.

Source of Variance	The Sum of Squares of Deviations	Degree of Freedom	Mean of the Sum of Squares of Deviations	The Value of F	Significance Level
Factor C	0.0732	2	0.0366	21.22	Yes
Factor D	0.0117	1	0.0117	6.81	Yes
Interactions of C and D	0.0096	2	0.0048	2.79	No
Error	0.0207	12	0.0017		
Total sum value	0.1152	17			

In conclusion, the ambient temperature has a significant impact on the adhesion strength of the frozen ice on the surface of the two materials (aluminum alloy or silicone rubber). The duration of freezing did not significantly affect the adhesion strength of frozen ice on the surface of both materials. The interaction of the ambient temperature and the freezing duration had a significant effect on the adhesion strength of the frozen ice on the aluminum alloy surface, but not on the frozen ice on the silicone rubber surface. Both the type of water quality and the type of material significantly affected the adhesion strength of the frozen ice, but the interaction between the two did not significantly affect the adhesion strength of the frozen ice.

Deficiency in the test: the deformation and strength of the ice are not isotropic and change with the thickness and lead orientation [23]. The reasons for the dispersion of some test results are complex. First of all, ice has a granular ice structure, that is, ice crystals are similar to particles, and its mechanical properties are isotropic. The other is a columnar ice structure, whose crystal morphology is different in the direction of the parallel ice surface and vertical ice surface, so the peak strength of horizontally loaded samples and vertically loaded samples are also different. That frozen ice is manually generated in a mold by a drip method is convenient to simulate the slow freezing process in nature, but has a certain probability to create a certain amount of anisotropy of columnar ice. However, the tensile shear direction of the ice was not distinguished during the test, which may be the reason for the large dispersion of the test results under some working conditions. Therefore, it is recommended to distinguish the direction of ice growth when performing mechanical properties tests such as the formation of frozen ice by drop method or the impact of ice, so that better test results may be obtained.

4.4. Analysis of Systematic Errors of the Test

When the specimen is shear inside the ice layer, the line of the tension is parallel to the shear surface, so there is no systematic error and the test results are accurate. When the ice layer of the adhesion surface falls off, there is a bias (1 mm) between the tension line and the adhesion surface, so the measured of the ice-breaking load is affected by the additional bending moment, so the system error needs to be analyzed.

Taking the test condition of distilled water freezing for 2 h at $-25\text{ }^{\circ}\text{C}$ as an example, the shedding of the adhesion surface of the specimen is caused by the combination of vertical shear force and additional bending moment.

The tensile force of the test:

$$\max\{F_0\} = 151\text{ N} \quad (4)$$

The thickness of ice:

$$\Delta = 2\text{ mm} \quad (5)$$

Adhesion area:

$$A = 622.25 \times 10^{-6}\text{ m}^2 \quad (6)$$

Additional bending moment:

$$M = F_0 \cdot \frac{\Delta}{2} = 0.151 \text{ N} \cdot \text{m} \quad (7)$$

Bending section coefficient:

$$W = \frac{bh^2}{6} = \frac{25 \times 25^2 \times 10^{-9}}{6} \text{ m}^3 = 2.604 \times 10^{-6} \text{ m}^3 \quad (8)$$

Bend positive stress:

$$\sigma_{t,max} = \frac{M}{W} = \frac{0.151}{2.604 \times 10^{-6}} \text{ Pa} = 0.058 \text{ MPa} \quad (9)$$

Check the tensile strength value of the frozen ice test designed by Xiao, Z. [21]:

$$\sigma_{t0} = 0.46 \text{ MPa} \quad (10)$$

Obtain:

$$\sigma_{t,max} \ll \sigma_{t0} \quad (11)$$

That is, the additional bending moment caused by the deviation of the tension line and the adhesion surface will not cause the ice damage, and the test can normally measure the shear adhesion strength on the adhesion surface.

5. Conclusions

By summarizing the data of this test and combining them with the freezing condition of the separation surface of the aircraft moving parts in the high and cold region, the following conclusions can be given:

(1) The adhesion strength of the frozen ice at the separation surface of the plane is studied, forming the test technology of metal/non-metal frozen ice adhesion strength. Moreover, the improved test parts have good freezing and test effects, and the new test operation is more rapid.

(2) The adhesion strength of the frozen ice of the aluminum alloy–ice–aluminum alloy increases first and then decreases with the decrease of the ambient temperature. The adhesion strength of the frozen ice of the aluminum alloy–ice–silicone rubber also increases first and then decreases along with the decrease of the ambient temperature. The frozen duration did not significantly affect the adhesion strength of the frozen ice on the surface of both materials. Due to the water quality, the adhesion strength of the frozen ice decreases in the order of distilled water, lake water and seawater, and the adhesion strength of seawater is only 1/22 of that of distilled water.

(3) The test technology of the adhesion strength of metal/non-metallic frozen ice can provide a reference for the test of other mechanical properties of ice. The test data of the adhesion strength of the frozen ice can provide data support for the determination of the ice-breaking load of the aircraft movement mechanism, the selection of the booster of the motion parts and the design of the anti-icing/deicing system of the aircraft.

Author Contributions: Conceptualization, X.X. and G.Q.; methodology, X.X.; validation, Y.F.; formal analysis, G.Q.; investigation, G.Q.; resources, X.X.; writing—original draft preparation, G.Q.; writing—review and editing, X.X.; supervision, Y.F.; project administration, T.L. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by China Aeronautical Science Foundation OF FUNDER grant number 20200009053002. The APC was funded by Aeronautical Science Foundation.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data is contained within the article.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Douglass, R.G.; Palacios, J.L. Effects of strain rate variation on the shear adhesion strength of impact ice. *Cold Reg. Sci. Technol.* **2021**, *181*, 103168. [[CrossRef](#)]
2. Work, A.H. A statistical analysis of impact ice adhesion strength data acquired with a modified lap joint test. In Proceedings of the AIAA AVIATION 2020 FORUM, Online, 15–19 June 2020.
3. Zou, M.; Beckford, S.; Wei, R.; Ellis, C.; Hatton, G.; Miller, M.A. Effects of surface roughness and energy on ice adhesion strength. *Appl. Surf. Sci.* **2011**, *257*, 3786–3792. [[CrossRef](#)]
4. Gagnon, R.; Bugden, A.; Garvin, M.; Teigen, S.; Elliott, B. *Ice Adhesion Shear Tests on Ice-Phobic Surfaces*; Lulea University of Technology: Lulea, Sweden, 2021; Volume 6.
5. Rnneberg, S.; Laforte, C.; Volat, C.; He, J.; Zhang, Z. The effect of ice type on ice adhesion. *AIP Adv.* **2019**, *9*, 055304. [[CrossRef](#)]
6. Rebekah, G.D.; Jose, L.P.; Grant, M.S. Design, Fabrication, Calibration, and Testing of a Centrifugal Ice Adhesion Test Rig with Strain Rate Control Capability. In Proceedings of the 2018 Atmospheric and Space Environments Conference, Atlanta, GA, USA, 25–29 June 2018; Volume 6, p. 3342.
7. Pervier, M.; Lerma, B.G.; Moncholi, E.P.; Hammond, D.W. A new test apparatus to measure the adhesive shear strength of impact ice on titanium 6Al-4V alloy. *Eng. Fract. Mech.* **2019**, *214*, 212–222. [[CrossRef](#)]
8. Matsushita, H. A Study on the Shearing Strength and Adhesive Strength of Sea Ice. *Mar. Dev. Proc.* **1997**, *13*, 765–770. [[CrossRef](#)]
9. Chu, M.C.; Scavuzzo, R.J. Adhesive shear strength of impact ice. *AIAA J.* **1991**, *29*, 1921–1926. [[CrossRef](#)]
10. Chen, T.; Cong, Q.; Sun, C.; Jin, J.; Choy, K. L. Influence of substrate initial temperature on adhesion strength of ice on aluminum alloy. *Cold Reg. Sci. Technol.* **2018**, *148*, 142–147. [[CrossRef](#)]
11. Zhang, Y.J.; Guo, R.Z.; Zhang, Y.H.; Liang, K. Adhesion shear strength test method for freshwater/seawater ice on carbon ceramicbrake pads of amphibious aircraft. *Aircr. Eng. Aerosp. Technol.* **2022**, *5*, 781–791. [[CrossRef](#)]
12. Jin, J.F.; Li, Y.; Chen, T.K. The effect of coating elastic modulus on icing adhesion strength. *J. Jilin Univ.* **2017**, *47*, 5.
13. Dawood, B.; Yavas, D.; Bastawros, A. F. Characterization of Ice Adhesion: Approaches and Modes of Loading. In Proceedings of the 2020 AIAA Aviation Forum, Virtual Event, 15–19 June 2020; Volume 6, p. 2802.
14. Prashanth, B.; Rye, W.; Hui, H. Ice Adhesion Measurements of Ice Mitigation Coatings Pertinent to Aircraft Icing. In Proceedings of the 9th AIAA Atmospheric and Space Environments Conference, Denver, CO, USA, 5–9 June 2017; Volume 6, p. 3928.
15. Pan, H. Modeling and Simulation of Aircraft Frozen Ice Form Prediction. *J. Syst. Simul.* **2014**, *1*, 221–229.
16. Zhang, Y.J.; Zhang, Y.H.; Guo, R.Z. Summary of the Test Methods for Icing Strength of Freshwater and Seawater. *J. Phys. Conf. Ser.* **2021**, *1786*, 012044. [[CrossRef](#)]
17. Dong, Y.Q. An application of Deep Neural Networks to the in-flight parameter identification for detection and characterization of aircraft icing. *Aerosp. Sci. Technol.* **2018**, *77*, 34–49. [[CrossRef](#)]
18. Susan Frankenstein, P.E.; Andrew, M.; Tuthill, P.E. Ice Adhesion to Locks and Dams: Past Work; Future Directions? *J. Cold Reg. Eng.* **2002**, *16*, 83–96. [[CrossRef](#)]
19. Aly, M.; Taylor, R.S.; Bailey, E.; Turnbull, I. Scale Effect in Ice Flexural Strength. *J. Offshore Mech. Arct. Eng.* **2019**, *141*, 051501. [[CrossRef](#)]
20. Janjua, Z.A. The influence of freezing and ambient temperature on the adhesion strength of ice. *Cold Reg. Sci. Technol.* **2017**, *140*, 14–19. [[CrossRef](#)]
21. Xiao, Z. *Reliability Study of the Brazilian Test Method for the Determination of Mechanical Properties of Ice*; Dalian University of Technology: Dalian, China, 2017.
22. Ding, L. Research on aircraft icing adhesion strength test. In Proceedings of the Eleventh National Academic Conference on Fluid Mechanics, Shenzhen, China, 3–7 December 2020.
23. Han, D.T.; Deborah, L.S.; Howard, L.S. An anisotropic elastic-decohesive constitutive relation for sea ice. *Int. J. Numer. Anal. Methods Geomech.* **2015**, *39*, 988–1013.