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Investigation of the Use of an Inorganic Aqueous Solution in Copper-Made Phase-Change Heat Transfer Devices

A novel inorganic aqueous solution (IAS) is shown to have a better heat transfer performance than water when used as the working fluid in copper-made phase-change heat transfer devices. First, the physical properties of IAS are measured and compared to those of water. Another, a chemical analysis is performed, and the chemical reactions involved between IAS and the copper surface are listed and categorized by their contributions to the heat transfer performance. In addition, a capillary rise test is performed to show how each chemical contributes to the improvement of the surface wettability. Last, using IAS in copper-made phase-change heat transfer devices is discussed, and the main focus is how IAS improves the heat transfer performance by a smaller thermal resistance and a larger critical heat flux. The conclusion is validated by thermo-siphon tests at different inclination angles. [DOI: 10.1115/1.4038190]

Introduction

Nowadays, with the rapid development of science and technology, the production and consumption of power has been increasing rapidly. As a result, thermal management has become a significant obstacle in almost every industry, such as electronics cooling of digital devices, waste heat recovery in large power systems, and nuclear power generation. The world market for thermal management products is predicted to grow from \$8.8 billion in 2013 to \$15.56 billion by 2018, reported by IRAP Inc. [1]. Innovative thermal solutions are demanded to keep the systems running at a peak efficiency and with a high reliability.

Phase-change heat transfer devices use evaporation and condensation of working fluids to transfer heat, and they have been a popular method of transferring heat for decades. Because the latent heat of evaporation is large, heat can be transported with a very small temperature difference between the heating source and the cooling source, which results in a high effective thermal conductance.

Water is one of the most popular working fluids used for the electronic cooling because of its large latent heat of evaporation. However, the performance of water cannot fulfill the needs of the rapid developments of science and technology, leading researchers to keep looking for methods of improving it. Wasekar and Manglik [2] added anionic surfactants to the working fluid to augment bubble incipience and increase the heat transfer coefficient. Wen and Ding [3] used nanoparticle suspensions to successfully increase the boiling heat transfer coefficient. However, it was found that overloading the solution with nanoscale particles can negatively impact the boiling performance (see Ref. [4]).

An inorganic aqueous solution (IAS), the subject of this work, was demonstrated to have a better heat transfer performance than water when used in copper-made phase-change heat transfer devices. A comparison of the performances of water and IAS was done by Reilly and Catton [5] in a vapor chamber test. A copper biporous wick was tested with water and IAS as the working fluid separately. It was found that IAS had a smaller thermal resistance and a later dry out compared to water in the vapor chamber test. How IAS works and the limitations of using IAS in different

copper-made phase-change heat transfer applications will be discussed in this paper.

What Is Inorganic Aqueous Solution

IAS fluid is an aqueous solution with the six inorganic chemical constituents shown in Table 1. A fresh IAS fluid is an aqueous solution with a dark red color (see Fig. 1(a)), and it has an initial pH number of 6.22. After being used in copper, aluminum, or stainless steel phase-change heat transfer devices, it becomes transparent, and the color turns to orange yellow, as shown in Fig. 1(b). The chemicals in IAS are inorganic and have much higher boiling points than water, so the vapor of IAS is pure water vapor (see condensed IAS vapor in Fig. 1(c)).

To reproduce the IAS fluid, any chemical compound which is a combination of the ions in Table 1 can be used as the source chemical. For example, a method of reproducing an IAS fluid is using potassium permanganate, potassium dichromate, chromium trioxide, calcium hydroxide, strontium hydroxide, and sodium hydroxide. Simply putting chemicals in de-ionized-water with overnight stirring will be sufficient. There is no redox reaction during the production process.

Physical Analysis. The physical properties of IAS at room temperature were measured and compared to those of water by UCLA. Table 2 is the summary of the comparison results. The density, boiling point, freezing point, surface tension, and viscosity of IAS are measured to be close to those of water. In addition, IAS is a very dilute aqueous solution. The mole fraction of the chemicals is 0.31%. Therefore, the thermal conductivity and specific heat of IAS should be very close to those of water [6].

Table 1 Chemical constituents in IAS

Positive ions	Negative ions
K ⁺	MNO ₄ ⁻
Na ⁺	HCrO ₄ ⁻
Ca ²⁺	Cr ₂ O ₇ ²⁻
Sr ²⁺	CrO ₄ ²⁻

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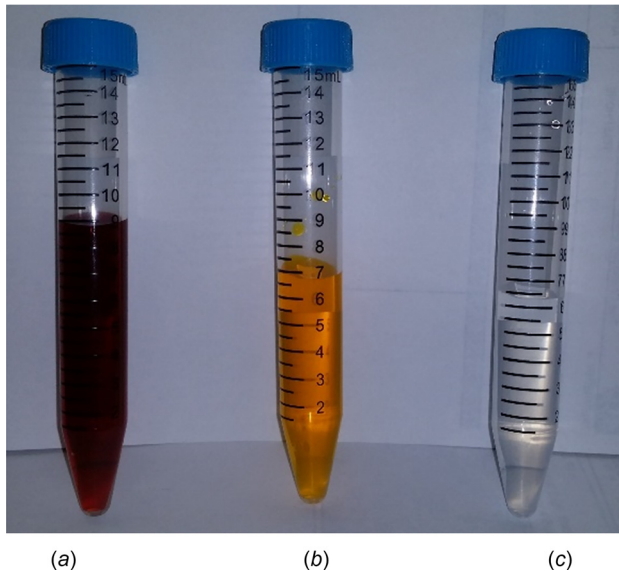


Fig. 1 (a) Fresh IAS, (b) used IAS, and (c) condensed liquid from the vapor of IAS

To sum up, the good heat transfer performance of IAS is not a result of changes of any bulk physical properties. In addition, after a copper surface is treated by IAS, a layer of coating is left on the surface, and DI-water has a much smaller contact angle on it, see Fig. 2(b) [7]. Even though the soluble part of the coating is washed off by DI-water, the surface still has a better wettability than the original smooth surface, see Fig. 2(c). As a result, the reactions between the chemicals in IAS and the metal surface change the surface wettability, and this should be the reason of the performance enhancement.

Chemical Analysis. The chemicals in IAS can be classified into three groups:

- (1) MNO_4^- , Cr (VI)
- (2) Ca^{2+} , Sr^{2+}
- (3) Na^+ , K^+

Based on this classification, a complete investigation of the involved reactions is done.

Water Self-Ionization. Water molecules can auto-dissociate and become hydrogen ions and hydroxide ions until an equilibrium is reached as shown by reaction (1)

Copper Substrate

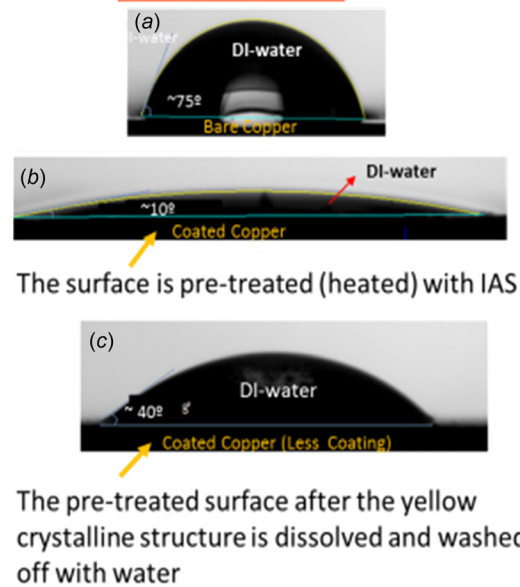


Fig. 2 Contact angle test on copper surfaces: (a) clean and smooth, (b) IAS treated, and (c) IAS treated and then rinsed by DI-water



Chromium (VI) Balance. Chromium (VI) exists in four forms in aqueous solutions: chromate acid, hydrogen chromate ion, chromate ion, and dichromate ion. With the pH number changing, the composition will become different (see reactions (2)–(4))

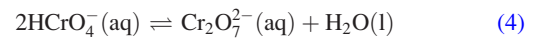
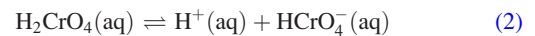


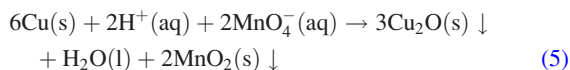
Figure 3 shows the relationship between the pH number and chromium (VI) concentrations in IAS. The existence of chromate acid can be neglected (<0.03%), when the pH number is larger than 4.

Table 2 Physical properties of water and IAS at 23 °C

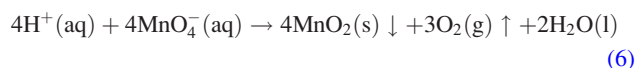
Properties	Water	IAS	Difference
Mass fraction of chemicals	0%	1.27%	+1.27%
Mole fraction of chemicals	0%	0.31%	+0.31%
Density	997.5 kg/m ³	1005.6 kg/m ³	+0.81%
Boiling point	100 °C	100.1 °C	+0.1 °C
Freezing point	0 °C	−0.3 °C	−0.3 °C
Thermal conductivity	0.604 W/m K	Mole fraction 0.31% Thermal conductivity change should be smaller than 1.3% [6]	
Contact angle on smooth copper surface	100 deg	98 deg	−2%
Surface tension	0.072 N·m	0.069 N·m	−4.2%
Dynamic viscosity	9.32×10^{-4} N s/m ²	9.60×10^{-4} N s/m ²	+3.0%
Potential heat of evaporation	2355.5 kJ/kg Average value from 70 °C to 100 °C	2350.0 kJ/kg Average value from 70 °C to 100 °C	−0.23%
Specific heat (c_p)	4.18 kJ/kg K	Mole fraction 0.31% Specific heat change should be smaller than 4.5% [6]	

Oxidation. There are two oxidizers in the IAS fluid: permanganate and chromium (VI). They may react with the metal surface based on the reductive ability of the surface material. Permanganate is a much stronger oxidizer than chromium (VI), so it can be seen to react with the surface first. Based on the pH number and the strength of the surface material as a reducer, chromium (VI) may or may not work as an oxidizer. For example, under neutral conditions, permanganate is the only oxidizer for copper surfaces, but both permanganate and chromium (VI) work as oxidizers on aluminum or iron surfaces [8].

When IAS is used as the working fluid in a copper phase-change heat transfer device, the copper surface reacts with the permanganate in IAS (see reaction (5)). Copper (I) oxide and manganese (IV) oxide will be generated and coat the surface. Moreover, the redox reaction consumes hydrogen ions, so the pH number goes up to 6.28 when permanganate is reacted out



However, over amount permanganate negatively affects the performance of phase-change heat transfer devices because of the oxygen gas, a noncondensable gas, generated by its disassociation (see reaction (6))



Hydrophilic Coatings. The vapor of IAS is pure water vapor, and one-dimensional diffusion model has built to demonstrate that advection is dominant in phase-change heat transfer devices [8]. Therefore, all the chemicals will be compressed to the meniscus region. Because of reaching solubility limit, it leads to coatings of surfaces in the evaporating region.

Reactions (7)–(9) show the three coatings which will be generated because of reaching the solubility limits. These salts will coat the surface with a porous structure, and the salts are hydrophilic. The porous hydrophilic coating will improve the surface wettability, provide extra capillarity, and enhance the heat transfer performance

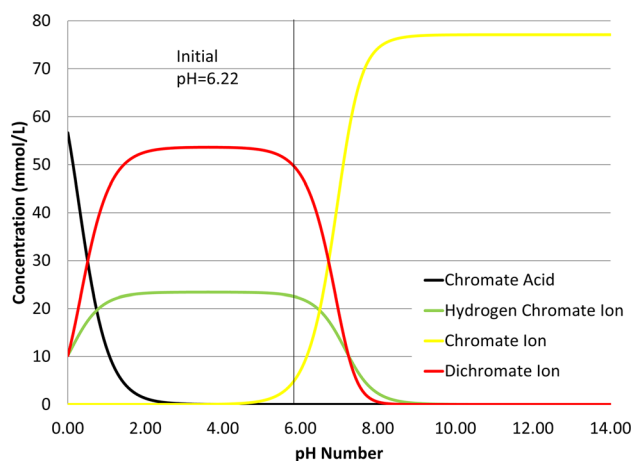
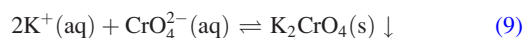
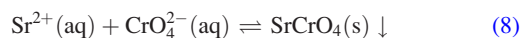
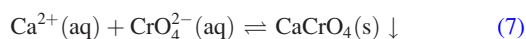


Fig. 3 Chromium (VI) balance in IAS at 25 °C

The sodium ion in IAS will not come out as its solid chromate salt because sodium chromate is a hygroscopic salt. It absorbs water vapor from the air to become a solution, so there is no solid sodium chromate generated until the water is completely evaporated out. Because of the strong bonding force between sodium chromate and water, it will be very difficult for the surface to be dried out. As a result, the surface is super hydrophilic during the operation, and the meniscus retreating, dry out, is largely delayed. This is an important part of the heat transfer enhancement.

Capillary Test

To understand how each type of coating contributes to the surface wettability, a capillary test was performed. This is only a visual test to show what happens in an operating phase-change heat transfer device, and it is not designed for heat transfer demonstration. Figure 4 is a schematic of the capillary test. One end of a mirror-like copper surface, 1.5 cm × 1.5 cm, was sandwiched by a cartridge heater block and a stainless steel plate, with the mirror-like side facing up. The opposite end of the surface was put into the liquid in a reservoir, and the angle between the surface and the horizontal is 45 deg. A digital microscope was used to monitor the capillary rise on the mirror-like side of the copper surface.

Four tests were done, and the solutions and surfaces used were listed as follows:

- DI-water and a clean mirror-like copper surface
- inorganic aqueous solution yellow and a clean mirror-like copper surface
- potassium permanganate solution, with the same amount of manganese as IAS, and a mirror-like copper surface
- inorganic aqueous solution yellow and a mirror-like copper surface pretreated by the potassium permanganate solution used in test (b) and then rinsed by DI-water

Inorganic aqueous solution yellow has the same chemical concentrations as IAS but no permanganate. During the test, 50 W was applied to the cartridge heater, and the temperature of the surface would be stabilized at 105–110 °C after 3 min.

Before starting capillary tests, a preliminary test was done. One droplet of IAS, potassium permanganate solution, and IAS yellow were put on three mirror-like copper surfaces separately. The surfaces were located on a hot plate with the plate temperature controlled at 105 °C, no bubbling. The surfaces were heated until the solutions were completely evaporated out.

Figure 5 shows the coated regions compared to the initially wetted ones. The black circles are the initially wetted regions. It shows that all the solid potassium chromate only coats within the initially wetted region. However, the wetted regions of potassium permanganate and IAS expand during the heating, and the final coated regions are much larger than the initially wetted regions. This phenomenon can be explained by the reaction between the permanganate ion and the copper surface, which changes the surface wettability. However, for chromate solutions, their contact

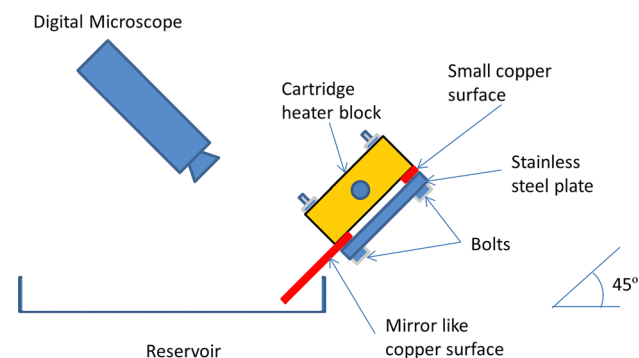


Fig. 4 Schematic of capillary test

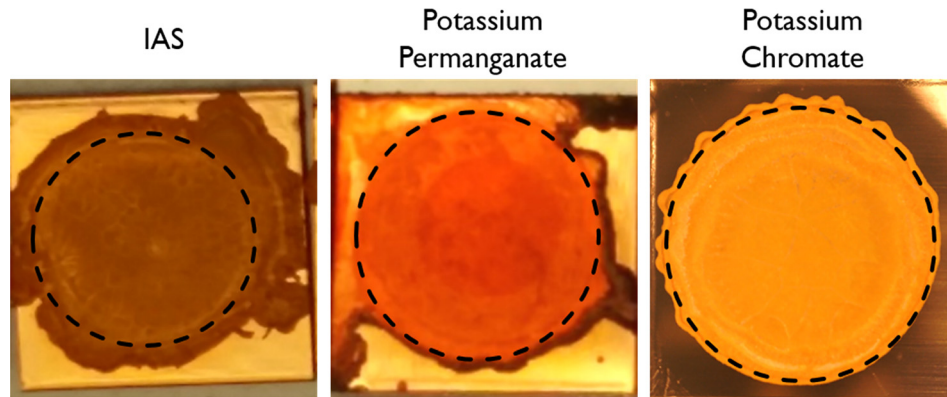


Fig. 5 Initially wetted region versus coated region

angles on clean copper surfaces are large and do not change with temperature increasing, so solid chromate salts are generated in the initial meniscus region and form a ring.

In fact, the redox reaction between the permanganate ion and the copper surface completes within an hour after the heat transfer device is charged. It should not cause meniscus creeping during operation. However, it opens the door to investigating the performance enhancement of IAS. A guess can be made that chromate solutions have a better wettability on permanganate pretreated copper surfaces.

Figure 6 shows the results of the capillary tests. For each test, a picture before heat is added and a picture after the surface temperature is stable are shown. The source light was adjusted to have a reflection on the meniscus. If the reflected light disappeared, it meant that the contact angle changed. For water and IAS yellow, the contact angle did not change, see Figs. 6(a) and 6(b). For IAS yellow, because of the generated solids piled up in the meniscus region, most of the bubbling occurred there and caused splashes.

For potassium permanganate solution, the contact angle changed during the test, see Fig. 6(c). The reflection of the source light completely disappeared after the surface temperature was stable. It was a result of the reaction between permanganate and the copper surface. The reactive surface made the contact angle smaller and pulled up the meniscus. However, the capillarity caused by this was limited, and the meniscus creeping on the horizontal surface was not seen.

Figure 6(d) shows the test results of IAS yellow and the mirror-like copper surface which was pretreated by the potassium permanganate solution and then rinsed with DI-water for 5 min. After

the pretreatment, the copper surface could be seen to have the oxides coating only. Therefore, the surface wettability will not be affected by the soluble chemicals. The reflection of the source light was not from the meniscus region before heat was added, so IAS yellow had a smaller contact angle, a better wettability, on the oxide-coated copper surface. With the temperature increasing, the meniscus started climbing up the copper surface along the edges and at the center of the surface.

The climbing of the meniscus was not the result simply of a rougher surface because it did not happen before heat was added. An explanation can be given that chromate solutions have smaller contact angles on the oxide-coated surfaces than those on clean copper surfaces, and their contact angles decrease as the concentrations increasing. Because of the evaporation, the chemical concentrations in the meniscus region increase, so the contact angle decreases. This causes the climbing up. In addition, because of reaching solubility limits, solid chromate salts start being generated in the meniscus region and increase the film thickness of the meniscus, which makes the meniscus further extend, see Fig. 7. The solid chromate salts, which are further away from the meniscus region, are redissolved in the liquid, and the new solid chromate salts are generated in the meniscus region and keep pushing the meniscus to extend until an equilibrium is reached.

Figure 8 shows the difference between water and IAS working fluids when the surfaces are close to being dried out. For water, when the surface is close to being dried out, the meniscus retreats, and surface is no longer cooled by evaporation, which leads to dry out. However, for IAS, the meniscus does not retreat because the solid chromate salts provide extra capillary forces to pump the

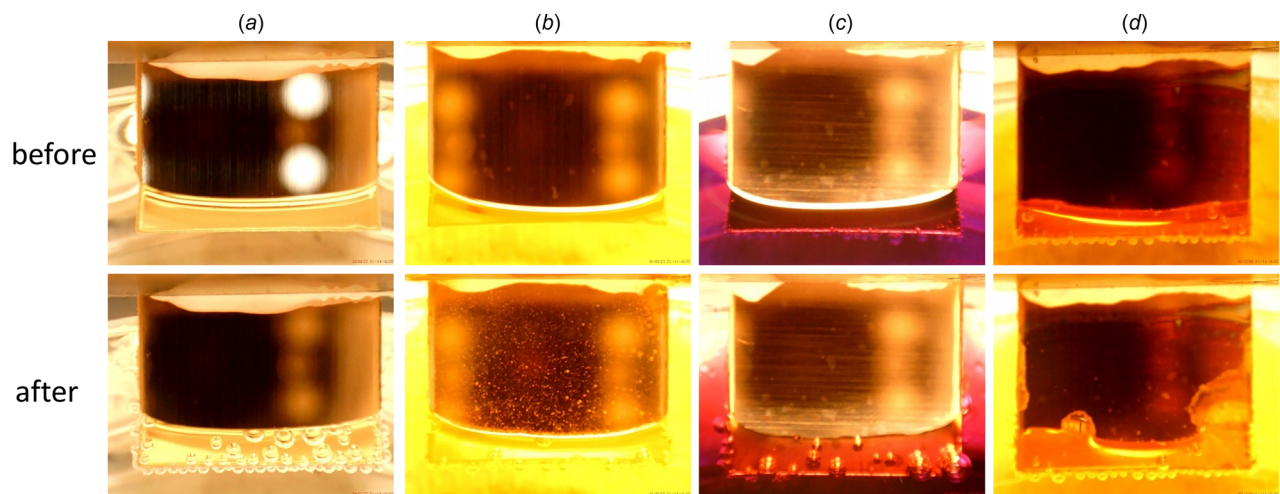


Fig. 6 Capillary test results: (a) water and smooth surface, (b) IAS yellow and smooth surface, (c) potassium permanganate solution and smooth surface, and (d) IAS yellow and surface pretreated by potassium permanganate solution



Fig. 7 Diagram of how chromate salts deposit on a copper surface pretreated by potassium permanganate

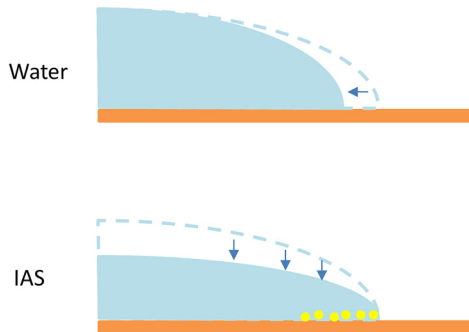


Fig. 8 Difference between water and IAS while being dried out

liquid to the meniscus region. Therefore, dry out is significantly delayed, and the liquid film just becomes thinner and results in an enhanced cooling.

In conclusion, it has been demonstrated that the combination effects of permanganate and chromate is the key to improve the heat transfer performance. The chromate solution has much better wettability on the surface pretreated by permanganate, and it will delay dry out and keep the device operating at higher heat fluxes.

Thermo-Siphon Test

A thermo-siphon test was developed to test the heat transfer performance of IAS at different inclination angles, and the results were compared to those of water. Copper thermo-siphons charged with DI-water, IAS yellow, and IAS were tested at four inclination angles: 90 deg (vertical), 30 deg, 20 deg, and 10 deg.

Experiment Setup. The copper thermo-siphon is made of a 45 cm long copper tube, with 3/8 in OD and 0.311 in ID. The tube

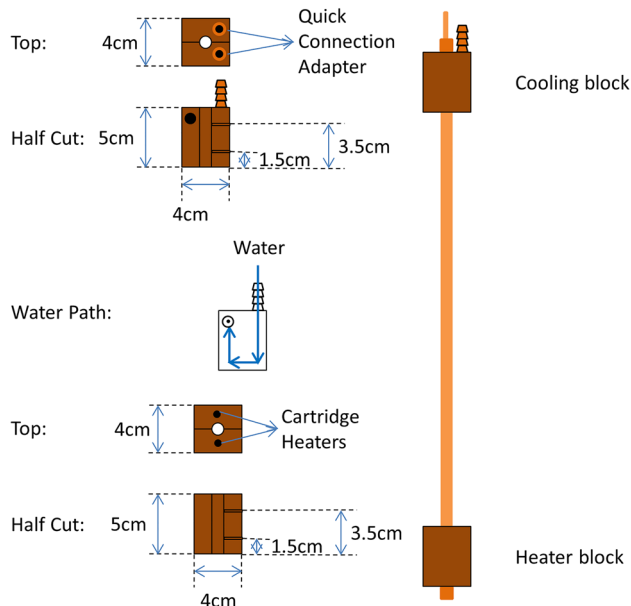


Fig. 9 Schematic diagram of the setup of the thermo-siphon experiment

is cleaned by fragment free wipes to remove dust and machine oil, and then pretreated by a 2 mol/L chloride acid solution to remove oxides. One end of the tube is sealed with a copper joint end cap and a 1/8 in OD copper fill tube by soldering. A 6 mm length copper rod, with 5/16 in OD, is punched into the other end of the tube and then sealed by soldering.

Before charging, the thermo-siphon is pretreated by the 2 mol/L chloride acid solution again to remove the oxide generated during the soldering, and then rinsed by DI-water for ten times. After being charged, the fill tube is sealed by cold welding. After being weighed, the sealing seam is protected by a J-B weld adhesive.

During the test, the thermo-siphon is heated by a cartridge heater block and cooled by a water cooling block with tap water, about 20 °C, see Fig. 9. The tap water is pumped through the cooling block by a peristaltic pump, with the flow rate 3.47 ml/s. The maximum input power that can be obtained is 320 W. As a result, the tests are initiated with an input power of 20 W for 15 min to reach a steady-state. Afterward, the input power is increased 20 W every 10 min until dry out or 320 W is reached.

Ten thermocouples are located on the outer surface of the tube: two in the evaporating region, two in the condensing region, and the other six in the adiabatic region (see Fig. 10). The temperatures data is collected by a PC with DASyLab.

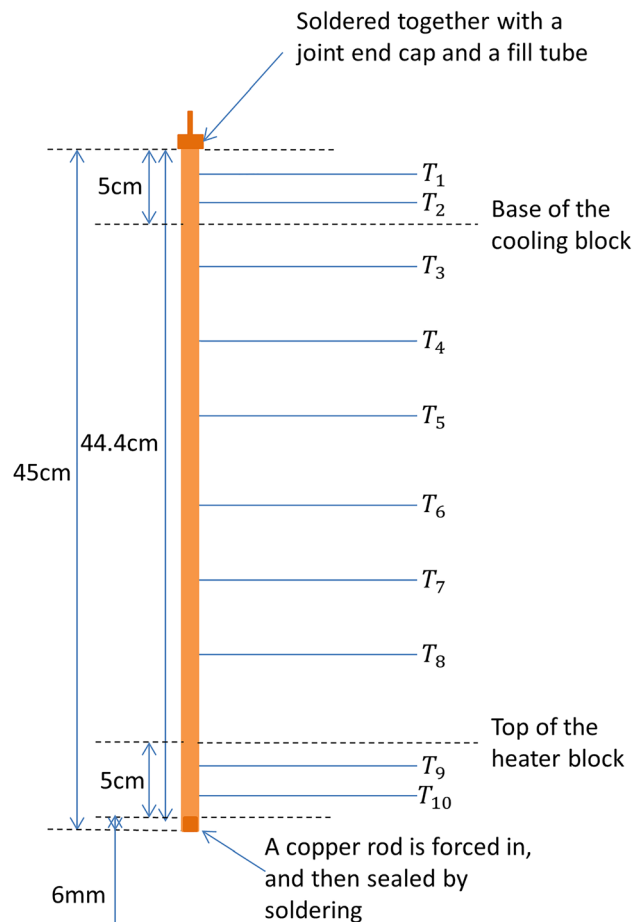


Fig. 10 Locations of thermo-couples in thermo-siphon test

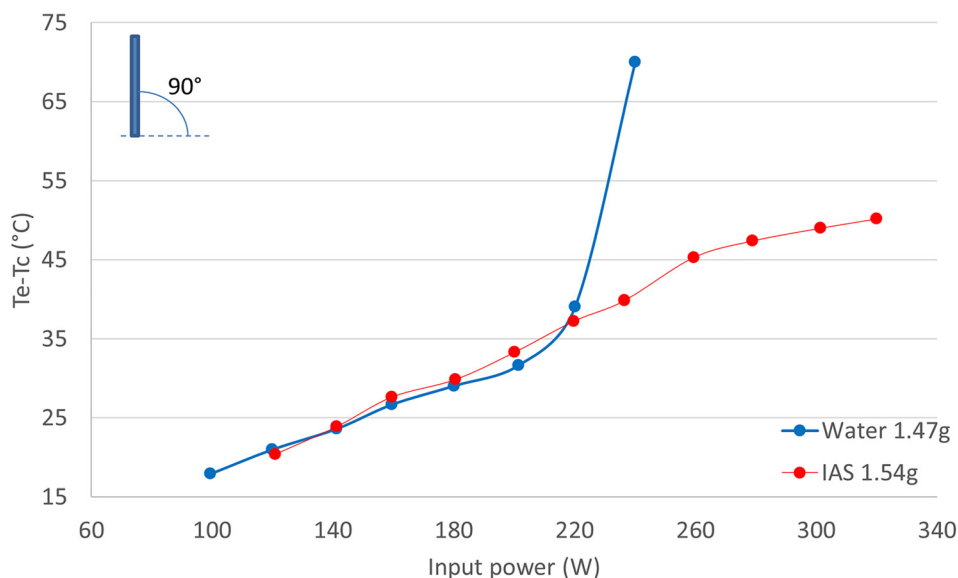


Fig. 11 Performance comparison between water/copper and IAS/copper thermo-siphons at an inclination angle of 90 deg

For the inclination angles of 90 deg and 30 deg from horizontal, about 1.47 ml working fluid, 60% of the volume of the evaporating region, was charged in the thermo-siphon based on the filling ratio given by Negishi and Sawada [9]. With the inclination angle decreasing, the effect of the gravity decreases, and more working liquid stays in the liquid back flow in the adiabatic and condensing regions. Therefore, about 3.60 ml of the working fluid, 16% of the total volume, was charged in the thermo-siphon tests for the inclination angles of 20 deg and 10 deg based on the filling ratio given by Faghri [10].

Test Results. Two runs were made for each thermo-siphon per inclination angle, and the results of the second runs will be used for comparison. In addition, when the temperature difference between the evaporator and the condenser is larger than 70 °C, the tube is assumed to dry out.

Figure 11 shows the performance comparison between water/copper and IAS/copper thermo-siphons at the inclination angle of

90 deg. The water tube starts working stably earlier and has a smaller temperature difference between the evaporator and the condenser below 220 W. After 220 W, the water-filled tube dries out, but the IAS-filled tube keeps working without drying out till the maximum input power 320 W.

Figure 12 shows the performance comparison between water/copper and IAS/copper thermo-siphons at the inclination angle of 30 deg. It is similar to that of 90 deg, but the water one dries out a lot earlier. The IAS one keeps working without drying out till 320 W.

Figure 13 shows the test results at an inclination angle of 10 deg. The water-filled thermo-siphon started working stably at 20 W, which was much earlier than the yellow and IAS-filled tubes, but it dried out at 100 W. The IAS yellow-filled tube started working stably at 80 W, but it dried out at 140 W. The IAS-filled tube could not reach the stable operation until 100 W, but it continued working without drying out until 260 W.

Figure 14 shows the test results for an inclination angle of 20 deg. The water thermo-siphon began stable operation the

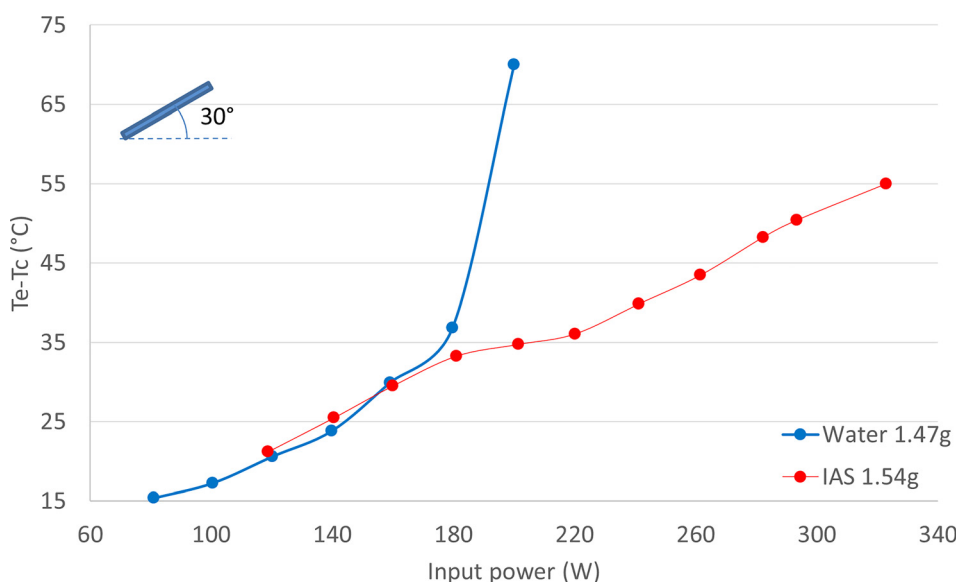


Fig. 12 Performance comparison between water/copper and IAS/copper thermo-siphons at an inclination angle of 30 deg

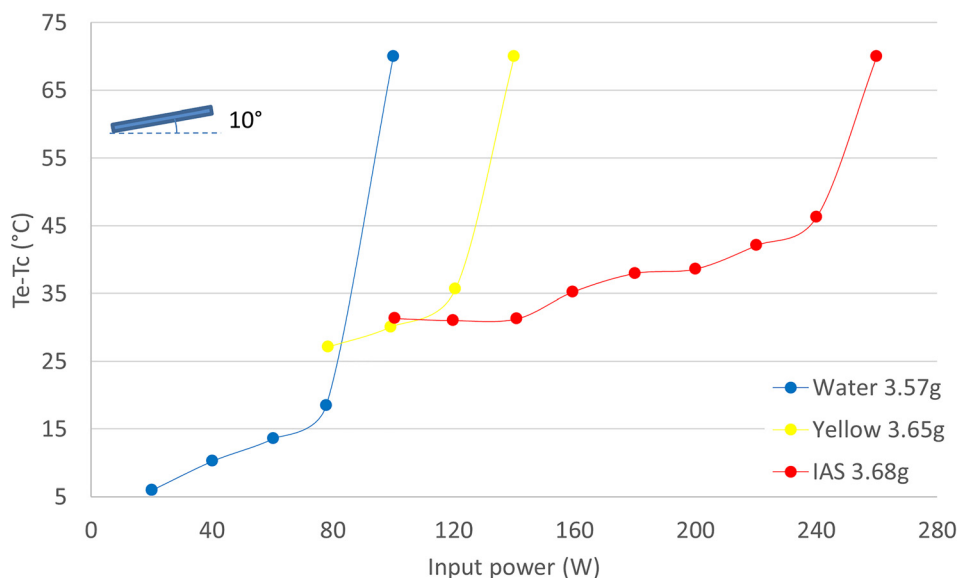


Fig. 13 Performance comparison between water/copper, IAS yellow/copper, and IAS/copper thermo-siphons at an inclination angle of 10 deg

earliest, but it dried out the earliest at 160 W. The yellow-filled tube dried out later than the water-filled tube, but not by very much. The IAS-filled tube began stable operation at 100 W, and it had the same temperature difference between the evaporator and the condenser as the water-filled tube at 100 W. In addition, the IAS-charged thermo-siphon did not dry out at the maximum input power, 320 W.

Discussion. Based on the results of the diffusion model and the capillary tests, IAS will coat the surface that is submerged with a layer of oxides that are permanent during the operation. Solid chromate salts are generated in the meniscus region because of the advection of the liquid flow. Both improve the surface wettability and enhance the bonding force between the liquid and the surface. The meniscus regions in vertical IAS/copper and water/copper thermo-siphons are shown in Fig. 15 if the liquid back flow is not considered.

However, the thermo-siphon is a closed system, and the condensed liquid flows back to the evaporator along the tube wall.

This makes the meniscus region of the IAS/copper thermo-siphon different. Figure 16 is the original test results of the 1.47 g water/copper thermo-siphon at the inclination of 90 deg, and it can be divided into four working regions: not working, unstable operation, stable operation, and dry out.

When the test is initiated, the liquid in the evaporator is heated up. The thermo-siphon will not start working until the vapor pressure difference between the evaporator and the condenser is large enough to pump the vapor flow. In addition, while the thermo-siphon is working at low input powers, the condensed liquid flows back to the evaporator by droplets. This causes the pulsatile temperature profiles in the unstable operation region. Moreover, the stable operation will not be reached until a stable film flow is formed. Last, when the boiling limit is reached, the thermo-siphon starts to dry out.

For an IAS/copper thermo-siphon, it follows the same working regions as the water one. While the difference is that before a film flow is formed, chromate salts will be generated and coat the meniscus region, as shown in Fig. 15. When a film flow is formed,

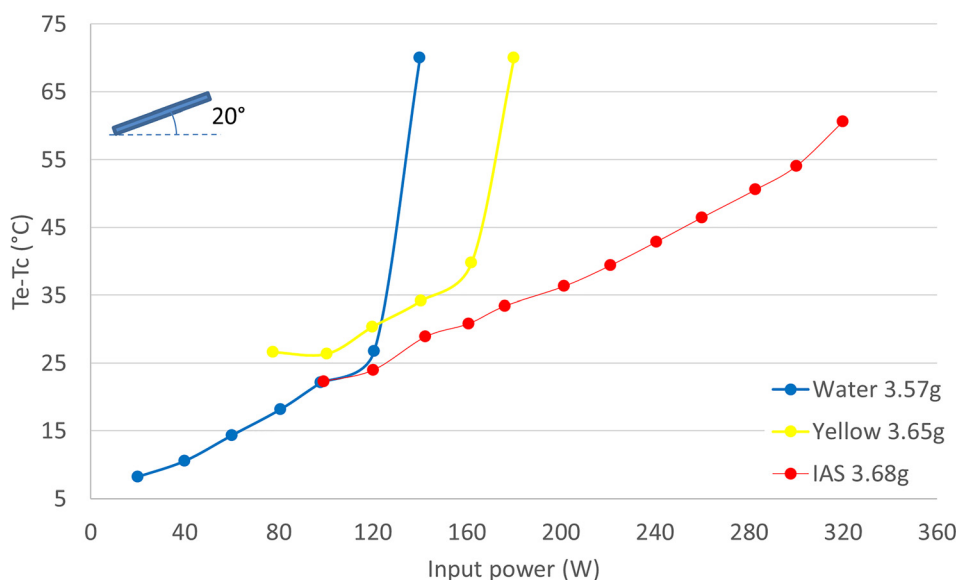


Fig. 14 Performance comparison between water/copper, IAS yellow/copper, and IAS/copper thermo-siphons at an inclination angle of 20 deg

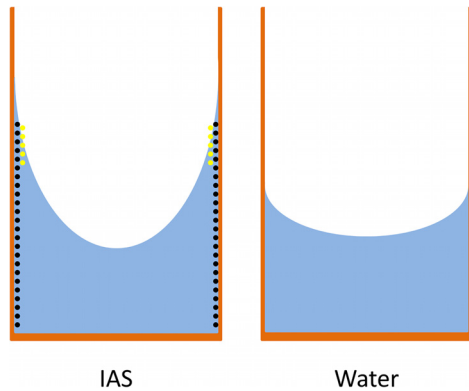


Fig. 15 Meniscus regions in vertical IAS/copper and water/copper thermo-siphons, without liquid back flow

it starts to wash off the previously generated chromate salts. The coatings bring extra resistance to the liquid flow and cause an extra instability, so the IAS/copper thermo-siphon reaches stable operation later than the water one. When the stable operation is reached, the chromate salts are washed off, and the film thickness of the liquid flow is large enough to submerge the oxides coating. In this case, the curvature of the liquid/vapor interface of the IAS thermo-siphon is the same as that of the water thermo-siphon, see Fig. 17.

As a result, for a given input power, if both IAS/copper and water/copper thermo-siphons, at inclination angles of 90 deg and 30 deg, are operating in the stable operation region, they should have similar heat transfer performances, similar thermal resistance. This can be demonstrated by the test results in Figs. 11 and 12.

For small inclination angles, the condensed liquid flows back to the evaporator only along one side of the tube (see Fig. 18). In region (1), where the meniscus is interrupted by the liquid back flow, the chromate salts are washed off, and the oxides coating is submerged by the liquid film, just like the case in Fig. 17. However, in region (2), the meniscus is not interrupted by the liquid back flow, as the case in Fig. 15, so both chromate salts and oxides coat the meniscus region and enhance the surface wettability. Therefore, an inclined IAS/copper thermo-siphon should have a better heat transfer performance, smaller thermal resistance, than the water/copper one.

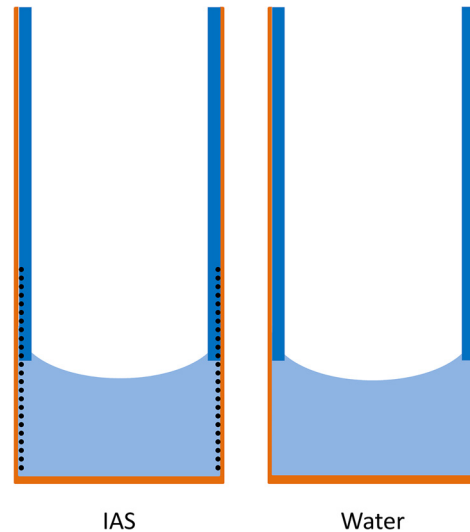


Fig. 17 Meniscus region in vertical IAS/copper and water/copper thermo-siphons, with liquid back flow

Because of the stable operation delay, the thermo-siphon test did not generate results to demonstrate the performance enhancement effect of IAS on a smaller thermal resistance, but this effect had been demonstrated by Supowit et al. [11] using a grooved flat heat pipe.

The thermo-siphon tests demonstrated the performance enhancement effect of IAS on delaying dry out. As mentioned in capillary test, the combined effects of the chromate solution and oxides improve the surface wettability, and it makes the meniscus more difficult to retreat. Even in case if the meniscus retreats, a layer of large solubility chromate salts will be left on the dried surface, and it makes the surface much easier to be rewetted. As a result, when nucleation boiling occurs, there will not be large bubbles generated, and the dried surface, caused by bubbling, will be rewetted immediately after the bubble departs. All above make boiling not a limit anymore to IAS/copper thermo-siphons.

However, IAS cannot delay the dry out of a device, the operational limit of which is not in the meniscus region, such as a sintered heat pipe with a thick monoporous wick. Nucleation usually initiates inside of the wick and close to the tube wall because it has the largest superheat. However, there is no chromate inside

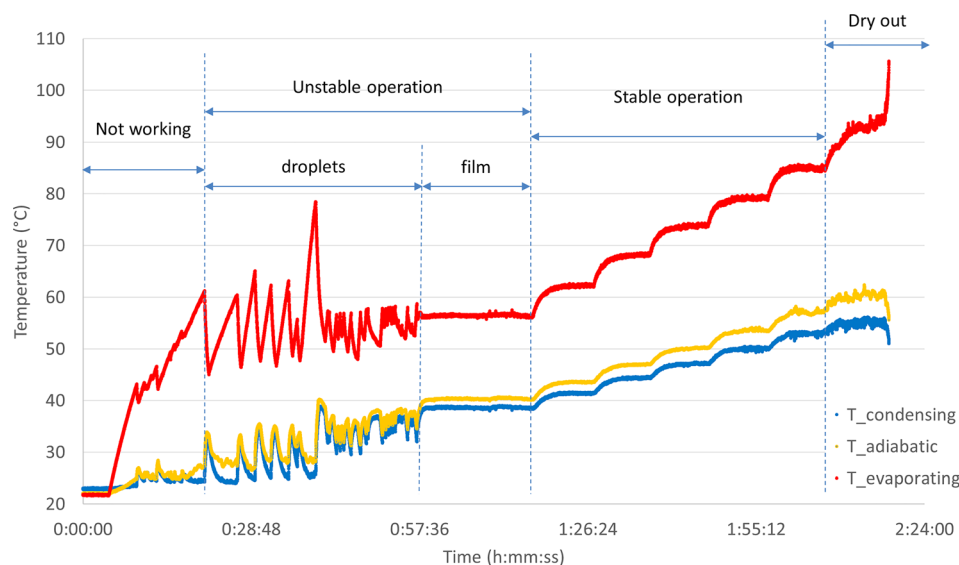


Fig. 16 Working regions of a thermo-siphon

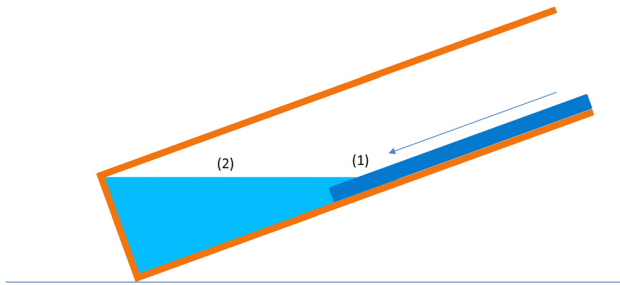


Fig. 18 Meniscus regions in an inclined thermo-siphon, with liquid back flow

the wick because of advection. Once bubbles are generated, the surface is dried out and will not be rewetted [8].

Conclusion

A new method of improving the heat transfer performance of phase-change heat transfer devices using chemical processes is introduced. When IAS is used as the working fluid in copper phase-change heat transfer devices, copper (I) oxide and manganese (IV) oxide permanently coat the entire submerged surface in the evaporator. Because advection dominates the diffusion of chemicals in the solution, all the soluble chemicals are pushed to the meniscus region. The high concentrated solution has a better wettability on oxide-coated surfaces. In addition, solid chromate salts are generated because solubility limits are exceeded and will coat the meniscus region with a porous structure to provide extra capillarity. These enhance the heat transfer performance and delay the dry out [12,13].

Future Work

The combination effect of oxides coating and chromate salts is the reason of the performance enhancement of heat transfer. However, over amount of chemicals will cause an extra thermal resistance or lead to clogging of the wick. Therefore, an optimal amount of chemicals requires further researches to find. The optimal amount should be a function of the total area of the meniscus region.

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