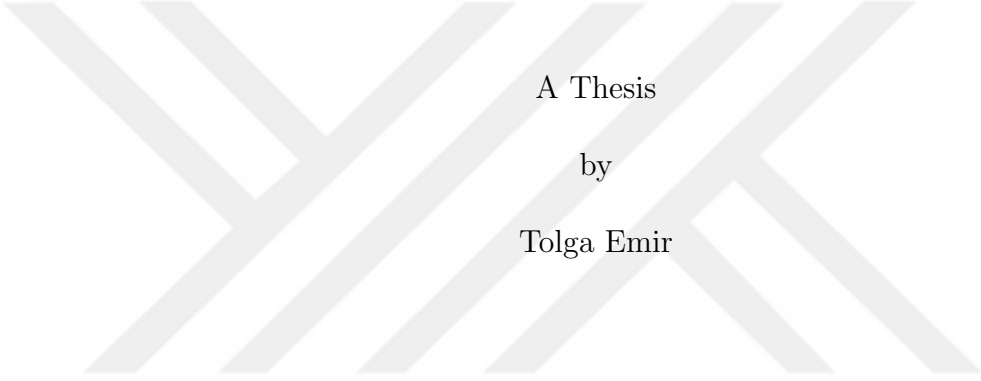


**EXPERIMENTAL INVESTIGATION ON POOL BOILING
HEAT TRANSFER OVER MICRO TEXTURED,
ADDITIVE MANUFACTURED SURFACES, AND
IMPACT OF EXTENDED EXPOSURE TO BOILING
HEAT TRANSFER**



A Thesis

by

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Submitted to the
Graduate School of Sciences and Engineering
in Partial Fulfillment of the Requirements for
the Degree of

Master of Science

in
Mechanical Engineering

Özyeğin University
August 2023

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HEAT TRANSFER**

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To my family and friends who trusted me...

ABSTRACT

The ever-increasing population and rising living standards require the development of high-performance, efficient, and safe technologies. Compact thermal systems face challenges due to limited space availability, while waste heat reduction and efficient cooling become crucial. Traditional thermal management methods have already reached their limits, and alternative technologies utilizing phase-change materials, heat pipes, or miniature/mini chambers are required to overcome heat removal limitations and achieve effective cooling in compact systems. Pool boiling heat transfer offers high-performance cooling opportunities for thermal problems especially electronics limited with high heat fluxes, and have been extensively studied over the last eight decades. This sensitive cooling method is influenced by a vast number of parameters, while the interplay of these parameters introduces a significant level of intricacy, making accurate predictions challenging. The predictive expressions for a broader spectrum of operating conditions, fluid chemical composition, or even surface topography are still inefficient in order to forecast the fundamental heat transfer measures of critical heat flux (CHF) and heat transfer coefficient (HTC). Indeed, this study aims to enhance boiling heat transfer (BHT) by incorporating artificial cavities through microdrill manufacturing and additive manufactured (AM) microchannel surfaces. Additionally, the development of oxide layers and their impact on surface performance are investigated. The boiling performance of surfaces with micro-drilled artificial cavities is analyzed under different operating conditions. In addition, the study examines the effect of microchannel geometries, specifically rectangular, V-shape, and inverse V-shape channels fabricated using laser powder bed fusion, on

BHT. The influence of surface roughness on heat transfer is compared between additive manufactured surfaces and conventionally polished surfaces. Oxide layer development and its effect on heat transfer are explored on copper substrates under various operating scenarios. In order to conduct the experiments, an in-house manufactured and fully automated boiling setup, which resist to high temperature and pressure was constructed. De-ionized (DI) water and 3M™ Novec™ hydrofluoroether (HFE-7100) Engineered Fluid were preferred as working liquids to be suitable for the cooling of electronic systems priorly. In this study, the tests were conducted at between 0 - 10 K subcooling temperatures and 1 - 1.5 bar pressures. Experimental analysis includes contact angle measurements, confocal microscopy, scanning electron microscopy, X-ray diffraction, and high-speed imaging for bubble formation. These investigations contribute to understanding and improving heat transfer performance in boiling systems.

ÖZETÇE

Hızla artan nüfus ve yükselen yaşam standartları, yüksek performanslı, verimli ve güvenli teknolojilerin geliştirilmesini gerektirmektedir. Sınırlı alan bulunması nedeniyle küçük ölçüdeki termal sistemler performans zorluklarıyla karşılaşırken, enerji fazlalığını azaltma ve verimli soğutma teknolojileri önem kazanmaktadır. Geleneksel soğutma yöntemleri kapasitelerinin sınırlarına ulaşmış durumda olup, faz değişimi bulunduran sistemler, ısı boruları veya minyatür/mini sıvı hazneler gibi alternatif teknolojiler, küçük ölçüdeki sistemlerin ısı transfer sınırlamalarının üstesinden gelmesi ve etkili soğutma sağlaması için önem arz etmektedir. 80 yıldır fizikçiler ve mühendisler tarafından yüksek dikkatle incelenen kaynama ile ısı transferi, yüksek ısı akımlarına sahip özellikle elektronik sistemler için performanslı soğutma fırsatları sunar. Bu hassas soğutma yöntemi birçok parametreden etkilenirken, bu parametrelerin etkileşimi doğru tahminleri zorlaştırır. Geniş aralıktaki çalışma koşulları altında, sıvının kimyasal bileşimi veya yüzey şekilleri gibi tahminsel ifadelerin temel ısı transfer ölçüleri olan kritik ısı akışı (CHF) ve ısı transfer katsayısı (HTC) için hala etkili tahmin yapamadığı koşullarda bu çalışma, yapay oyukları oluşturan mikro matkap üretimi ve katmanlı imalat metodu (AM) ile üretilmiş mikrokanal yüzeylerine dahil ederek kaynama ısı transferini (BHT) geliştirmeyi amaçlamaktadır. Buna ek olarak, yüksek sıcaklıklar geçiş metalleri üzerinde oluşması muhtemel oksit tabakalarının gelişimi ve yüzey performansına etkisi incelenmektedir. Mikro matkaplarla delinmiş yapay boşluklara sahip yüzeylerin kaynama performansı analiz edilmektedir. Ayrıca, toz yatak füzyon birleştirme kullanılarak üretilen dikdörtgen, V şekli ve ters V şekli kanalların mikrokanal geometrilerinin kaynama ile ısı transferi üzerindeki etkisi incelenmektedir. Yüzey pürüzlülüğünün ısı transferine etkisi, eklemeli üretilen yüzeyler ile

geleneksel olarak cilalanmış yüzeyler arasında karşılaştırılmaktadır. Oksit tabakası oluşumu ve ısı transferine etkisi, çeşitli çalışma senaryolarında bakır numunelerde araştırılmaktadır. Deneylerin yapılabilmesi için kendi bünyemizde geliştirilmiş tam otomatik yüksek sıcaklık ve basınca dayanıklı kaynatma düzeneği yapılmıştır. Önceleri elektronik sistemlerin soğutulmasına uygun çalışma sıvıları olarak deiyonize (DI) su ve 3M™ Novec™ hidrofloroeter (HFE-7100) özel sıvısı tercih edildi. Bu çalışmada testler 0-10 K aşırı soğutma sıcaklıklarında ve 1-1,5 bar basınçlarda gerçekleştirilmiştir. Deneysel analizler, temas açısı ölçümleri, konfokal mikroskop ile görüntüleme, taramalı elektron mikroskopu ile görüntüleme, X-ışını difraksiyonu ile görüntüleme ve kabarcık oluşumunu saniyede yüksek hızlı görüntüleme gibi yöntemleri içermektedir. Bu çalışmalar, kaynama sistemlerinde ısı transfer performansını anlamak ve geliştirmek için katkıda bulunmaktadır.

ACKNOWLEDGEMENTS

I would like to express my heartfelt gratitude to my thesis advisor, Prof. Dr. Mehmet Arık, for providing me with the opportunity to conduct my master's study under his guidance. His unwavering commitment, guidance, and constant support, even amidst the several challenges. His vision and the valuable experience gained under his supervision have been instrumental in my professional development and an honor to be his student, and the knowledge, motivation, and perspective I gained while working at the EVATEG Center will serve as a lifelong inspiration. I am thankful for the generous opportunities he has given me to contribute and grow.

I am immensely grateful to Assoc. Prof. Dr. Mete Budaklı, who played a pivotal role in my academic journey. Beyond being a mentor, he treated me like a beloved elder brother and guided me with unwavering support. His fatherly care, personal interest, and dedication to my development have been invaluable. He generously invested his time, energy, and extensive technical expertise, pushing me to achieve success. His meticulous planning and belief in my potential were instrumental in my accomplishments. I will forever cherish the special bond we shared, and I am deeply thankful for his guidance, mentorship, and the profound impact he had on my life.

I would like to express my deepest gratitude to my esteemed professors, Assoc. Prof. Dr. Özgür Ertunç and Assist. Prof. Dr. Altuğ Melik Başol, for their unwavering mentorship and guidance throughout my academic journey. I would also like to extend my appreciation to Gökhan Kayansalçık for his assistance with LABVIEW integration and access to production facilities. I am grateful to Hamza Ouarabi, a former team member, and Alper Saygın, my closest friend during my master's degree,

for their constant support and encouragement. Additionally, I would like to express my heartfelt thanks to my first mentor, Dr. Thamer Khalif, for introducing me to the laboratory and helping me adapt to the research environment. I surely special thanks go to my dear friends for their unwavering presence and support in all aspects of my life. Your companionship has made everything easier for me.

I am also thankful to the Scientific and Technological Research Council of Turkey (TÜBİTAK) and all members of Özyeğin University who I know for their partial support during my research. Thanks to Prof. Dr. Nima Shamsaei and Paul Mikyle for the use of the infrastructure of the National Center for Additive Manufacturing Excellence. In addition, for the surface analyses, I want to thank to Kadir Sağır from Turkish-German University and Dr. Hande Öztürk.

Last but not least, none of this would have been possible without the love, trust, and support of my family. I am eternally grateful for their encouragement in pursuing my interests and for allowing me to chase my dreams. Thank you for always believing in me.

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Symbols

q''	:Heat flux [W/cm ²]
HTC	:Heat transfer coefficient [W/cm ² K]
k	:Thermal conductivity [W/cmK]
R_a	:Average roughness [μ m]
T	:Temperature [°C]
x	:Distance [m]
D_b	:Bubble depature diameter [m]
V_{max}	:Maximum volume [m ³]
L	:Capillary length [m]
R_c	:Cavity radius [m]
g	:Gravity acceleration [m ² /s]

Greek symbols

Δ	:Difference
σ	:Surface tension [N/m]
ρ	:Density [kg/m ²]
ϕ	:Contact angle [°]

Subscripts

cal	:Calibration
$cbath$	:Calibration bath
$conv$	:Convection
$hole$	:Thermocouple position in holes
i	:Instantaneous
l	:Liquid

TC	:Thermocouple position
v	:Vapor
w	:Near the wall
$wall$	:Wall
w	:Saturation
1, 2, 3	:Thermocouple number
∞	:Bulk-liquid

Abbreviations

AM	:Additive manufactured/ing
BHT	:Boiling heat transfer
NBHT	:Nucleate boiling heat transfer
CA	:Contact angle (static)
CHF	:Critical heat flux
CNC	:Computer numerical control
DC	:Direct current
DI	:De-ionized
HFE	:Hydrofluoroether
HTC	:Heat transfer coefficient
MD	:Microdrilled
ICDD	:International Centre for Diffraction Data
IVR	:In-vessel retention
L-PBF	:Laser powder bed fusion
LM	:Lathe machined
PEEK	:Polyether ether ketone
SEM	:Scanning electron microscope
XRD	:X-ray diffraction

Symbols and superscripts

○	:Reference CHF value
●	:Reference CHF* value
□	:Reference value for the CHF** performances
*	:After 1 st 24h continuous boiling process
**	:After 2 nd 24h continuous boiling process
⊕	:Available analysis for SEM
⊕⊕	:Available analyses for SEM and XRD
+	:Performed experiment

CHAPTER I

INTRODUCTION

The ever-increasing population and rising living standards require the development of the technology, which delivers high performance, efficiency, and safety [1]. To address these requirements, ongoing technological advancements have been dedicated to achieve high power generation. In the meantime, restricted spatial constraints bring notable challenges for compact thermal systems such as denser thermal power generation. Indeed, reducing the amount of waste energy during system operations has been one of the major challenges in engineering, especially in areas where an excessive amount of heat is dissipated such as supercomputers, microelectronics, spacecraft avionics, nuclear reactors, and high-capacity electric batteries as given in Figure 1 [2, 3, 4]. Besides, concerning latter devices, the systems are further required to occupy small spaces as working speed and technical functionality have increased remarkably over the last few decades. Simultaneously, material selection as well as overall configuration, can lead locally to high temperatures when the cooling performance of thermal management is not sufficient. This phenomenon can either cause a malfunction or even damage system components. It is well-known that current thermal management systems using single-phase natural or forced convection have reached their heat removal limits. Thus, some technologies working with the principle of latent heat, such as phase-change materials, heat pipes, or miniature/mini cooling chambers, must replace single-phase cooling mechanisms. Pool boiling, a longstanding and efficient cooling method, offers high heat dissipation rates through the liquid-to-vapor phase change process [5, 6]. By employing this cooling technique, it becomes possible to minimize the reliance on fans, pumps, and other components that can hinder overall

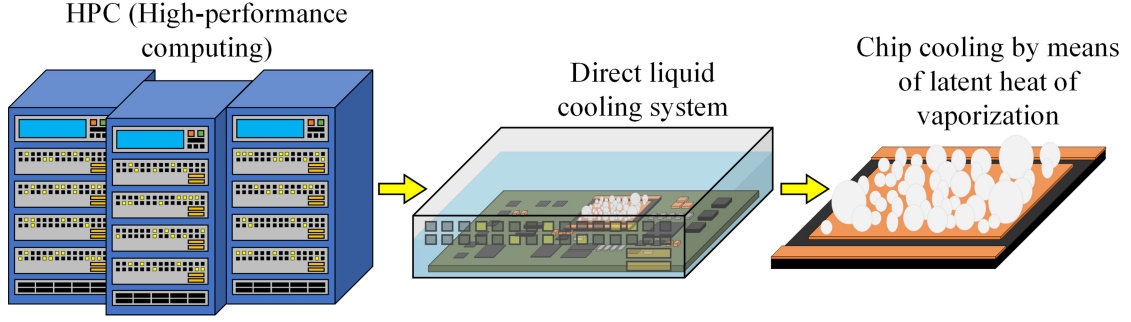


Figure 1: Exemplary application of direct liquid cooling system with phase-change cooling process: high-performance computing (HPC).

performance through pressure loss. Furthermore, it allows engineers to design systems that are more compact, reliable, and with a rather quiet operation ability.

1.1 Pool Boiling Fundamentals and Parametric Effects

Boiling is a phase-change phenomenon and occurs at the solid-liquid interface with a solid surface maintained at a temperature (T_s) higher than the saturation temperature (T_{sat}) of the liquid. The boiling mechanism is characterized by the rapid formation and growth of vapor bubbles within the liquid. This chaotic process encompasses intricate fluid motion patterns dominated by numerous thermophysical variables [7]. Heat transfer from the solid surface to the liquid during boiling follows the principles of Newton's law of cooling, which states that the rate of heat transfer between a solid surface and a surrounding fluid is directly proportional to the temperature difference between the surface and the fluid (T_∞). The law can be expressed as Eq. 1:

$$q'' = HTC(T_s - T_\infty) = HTC\Delta T_{superheat} \quad (1)$$

where $\Delta T_{superheat} = T_s - T_\infty$ is called the superheat temperature or excess temperature, which represents the temperature difference between the heating surface and the temperature of the fluid. Boiling can happen either under subcooled or saturated conditions, when the surface tension disappears at the critical more than the

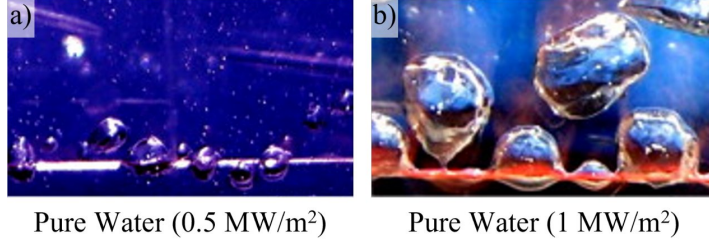


Figure 2: Pool boiling on metal wire in pure water at different heat fluxes of 0.5 MW/m² and 1 MW/m² [10].

saturation temperature. Boiling is a non-equilibrium process where bubbles formed during the rapid interface between vapor and liquid are not in thermodynamic equilibrium with the surrounding liquid. Heat transfer during boiling generally occurs through two mechanisms: bubble growth by absorbing energy from the liquid during evaporation and micro-convection heat transfer around the bubbles induced by the evaporation momentum force [8]. This complex process involves cycles of bubble growth and collapse, as well as the movement of liquid near the bubble interface, enhancing heat transfer.

In 1934, Nukiyama [9] made a groundbreaking contribution to the understanding of boiling and evaporation by conducting several experiments on heated nichrome and platinum wires immersed in liquids similar to the study [10]. He proposed four distinct boiling regimes as depicted in Figure 3: natural convection boiling to point A, nucleate boiling (A-C), transition boiling (C-D), and film boiling after point D, which are depicted on the classical boiling curve plotting the heat flux against the superheat temperature as illustrated in Figure 2. As the superheat increase, bubble inception eventually occurs and is defined as the onset of nucleate boiling (ONB) at point A in Figure 3, where the fluid motion can be predictable by free convection effects as turbulent on large horizontal plates with the following equation (Eq. 2):

$$Nu_L = 0.15Ra_L^{1/3} \quad (2)$$

where the Nu_L is Nusselt number and Ra_L is Rayleigh number. The vapor is released in the form of jets or columns, which subsequently merge into vapor slugs when the heat flux is increased. The close proximity of these densely packed bubbles can impede the movement of liquid near the surface. The coalescence of lateral bubbles and their departure from the surface result in the rapid increase of surface temperature and the formation of an instant vapor-insulating layer. As the heat flux continues to rise, the process of bubble formation and coalescence intensify while the rewetting of the surface becomes less efficient, leading to a decrease in the heat transfer coefficient (HTC). Eventually, at a certain heat flux level, the liquid supply to the heating surface, sustained by the instantaneous pressure from the bubble departures, becomes insufficient to replenish the surface with fresh liquid, resulting in the formation of a vapor cushion. This phenomenon is known as critical heat flux (CHF) [11, 12]. The performance of pool boiling is characterized by two essential measures, heat transfer coefficient (HTC) and critical heat flux (CHF). These essential quantities play a vital role in industrial designs and applications; otherwise, failure to transfer heat can result in performance limitation or catastrophic damages to system components, for instance, printed circuit board (PCB) components and In-Vessel Retention (IVR) in nuclear power plants [13, 14, 15]. Thus, many researchers have been investigating and developing distinguished methods to enhance and predict the pool boiling heat transfer (BHT) for more than 80 years [16, 17].

Various parameters directly influence this underlying sensitive heat transfer mechanism and bubble dynamics at different levels. Thus, many researchers have published a large number of correlations to predict bubble frequency, bubble departure diameter, overall heat transfer coefficient, and critical heat flux according to potential operation ranges controlled by several parameters. In common, experimental results show good agreement with Rohsenow [18] heat flux prediction (Eq. 3) and CHF predictions developed based on the original correlations by Kutateladze [19] in 1951 and Zuber

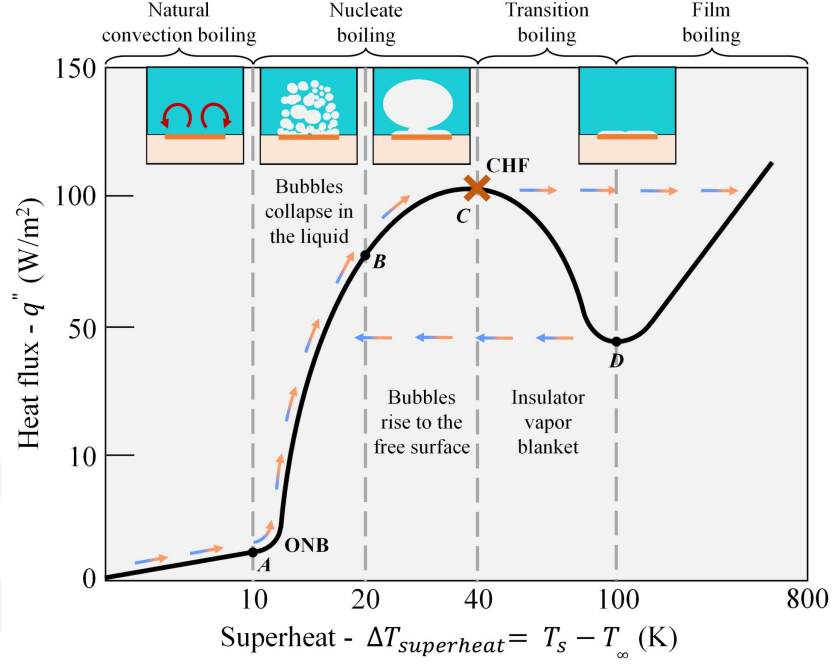


Figure 3: Nukiyama's boiling curve for saturated water at atmospheric pressure [9].

[20] in 1959 (Eq. 4). However, those correlations are valid for the limited conditions, which means predictive expressions for a broader spectrum of operating conditions, fluid chemical composition, or even surface topography can still be developed in order to forecast heat transfer parameters such as CHF, HTC , etc. [19, 21].

$$\frac{q}{A} = \mu_l h_{lv} \left[\frac{g \Delta \rho}{\sigma} \right]^{1/2} \left[\frac{c_{pl} (T_s - T_{sat})}{C_{sf} h_{lv} Pr_l^n} \right]^3 \quad (3)$$

$$q''_{CHF} = \frac{\pi}{24} p_v^{1/2} h_{lv} [g \sigma \Delta \rho]^{0.25} \quad (4)$$

To form a bubble during boiling, certain conditions, which include the presence of a small cavity or crack filled with gas on the heated surface and the surrounding fluid being heated to a high enough temperature, must be met. Before bubble growth starts, the bubble is in a state of thermostatic equilibrium, referring to a condition in which the bubble and its surroundings are at the same temperature. In this state, there is no heat transfer between the surroundings and the bubble, while its inside

temperature is uniform. As the bubble growth is concerned, the heat transfer mechanisms of conduction and convection play a leading role in the bubble size, shape, and temperature distribution. The bubble departure diameter (D_b) was first predicted by Fritz [22] in 1935 with the well-known correlation

$$D_b = 0.0208\theta\sqrt{\frac{\sigma}{g\Delta\rho}} \quad (5)$$

In addition, the maximum volume of the bubble (V_{max}) was presented by the expression

$$\frac{V_{max}}{L^3} = \varphi(\theta) \quad (6)$$

where the L is the capillary length scaling factor between gravity and surface tension

$$L = \sqrt{\frac{2\sigma}{g\Delta\rho}} \quad (7)$$

On the other hand, the bubble nucleation can be separated according to formation locations such as artificial cavities instead local imperfections/micro-roughness on the heated surface. Indeed, the maximum and minimum cavity radius (R_c) for initiating a bubble growth was first introduced by Han and Griffith [23, 24] in 1965 as depicted in Eq. 8 (see Figure 4).

$$R_c = \frac{\delta(T_w - T_{sat})}{3(T_w - T_\infty)} \left\{ 1 - \frac{12(T_w - T_\infty)T_{sat}\sigma}{(T_w - T_{sat})^2\delta\rho_v h_{lv}} \right\} \quad (8)$$

Cavities that fall outside of this range are not considered viable nucleation sites. This can be explained with the cavity with a radius exceeding (R_c) will eventually become inactive as the surrounding fluid invades it. This occurs because the gas pressure inside the cavity is insufficient to maintain a stable equilibrium with the

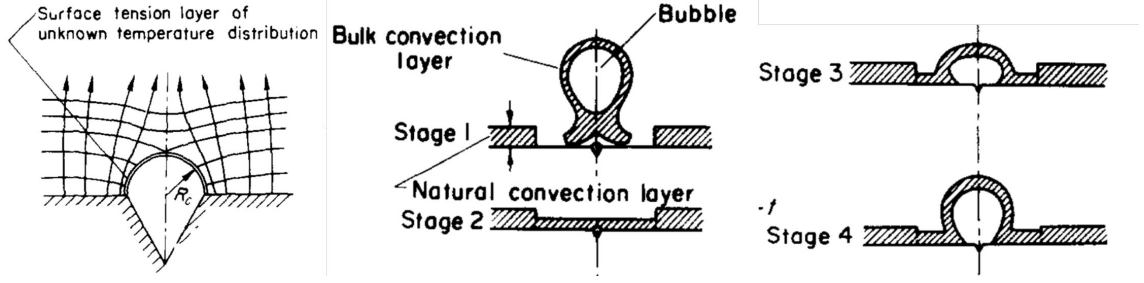


Figure 4: Temperature of fluid and bubble growth near a heating surface [23, 24]

surface tension of the fluid. As a result, the cavity loses its ability to support bubble formation. In addition, multiple research articles address the calculation of the bubble departure diameter, taking into account additional factors such as heat flux, superheated thermal layer (Zuber [20]), or the enthalpy of evaporation (Cole [25]), which offers valuable insights. At least, Chesters [26] presented the shape and size of the bubble formed on a sharp- or round-edged orifice with the help of analytical solutions in 1977. Two modes of formation were distinguished depending on the natural contact angle (ϕ). Accordingly, the bubble is confined to the orifice (ϕ small) or the bubble spreads beyond the orifice (ϕ large: Fritz) as shown in Figure 5. Once the growth occurs according to mode A, the scale of the bubble profile reaches maximum as the attachment plane reaches to the point N as depicted in Figure 5. Opposite to mode A, mode B growth interested in the bubble growth in small orifices and agrees with the well-known numerical result of Fritz [22]. In conclusion, once the mode A considered, the maximum volume of the bubble is seen to be given as follows:

$$V_{max} = \frac{2\pi R_c \sigma}{g\rho} \phi \quad (9)$$

The parametric effects significantly control the heat transfer coefficient and critical heat flux of the thermal systems. In addition to heat flux stating the surface temperature, the parameters are formed by the system pressure, subcooling temperature, surface topography, surface orientation, working fluid, and combined effects for the plain heaters. In those, pressure induces the CHF and HTC with smaller

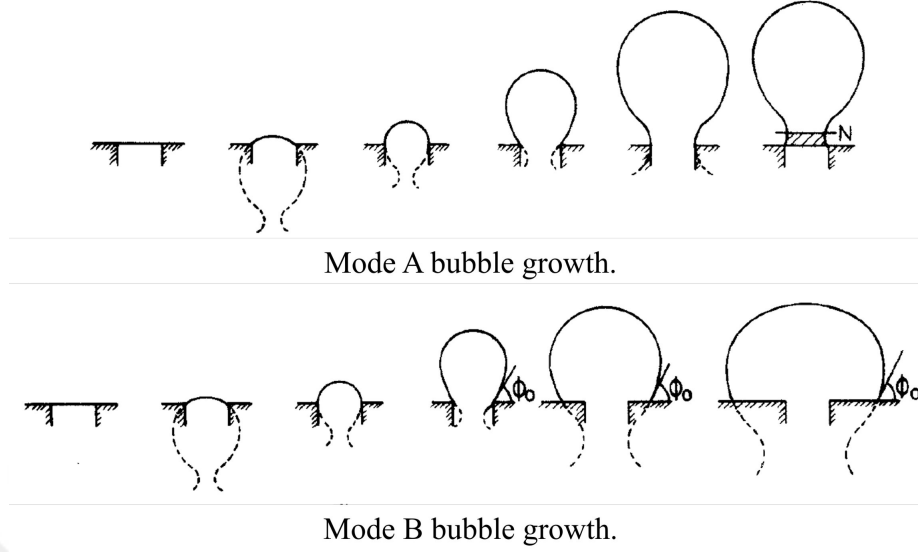


Figure 5: Stage of bubble growth due to an instability of the profile to small perturbation in different modes of A and B [26].

bubble departures and intensified activation of surface cavities, even the surface heat flux increases. The optional parameter, which is subcooling decreases bubble generation and diameter, hindering vapor bubble traceability. Heater size and surface topography influence nucleate boiling, and regulating wettability, thermocapillary, and instability of the surface size influence the nucleation site density and capillary suction positively. Surface orientation impacts CHF, with downward-facing surfaces showing an exponential decrease. Fluid properties, including nano-fluids and binary mixtures, significantly affect heat transfer, but excessive nanoparticles and ineffective mixtures have a great potential to dramatical decrease on boiling performance. As a result of broad and deep discussions, the effect of surface topography steps forward with its from many other abovementioned parameters [21]. Furthermore, it is concluded that surface topography can be artificially modified either mechanically, chemically, or biologically to enhance nucleation site density, surface wettability, and capillary pressure on the heating surface [27, 28].

1.2 *Methods for Heat Transfer Enhancements*

Once the developed insightful methods enhancing BHT and the correlations in particular conditions are considered, many studies reported inconsistencies in the enhancement of heat transfer trends, specifically for surfaces modified through chemical and biological treatment [29]. As these developments continued, most of the studies did not discuss in detail the impact of aging in long-term operations and repeatability because of possible performance change on the latest applications [30]. Hence, the uncertainty of the critical measures, CHF and HTC , for boiling surfaces can raise doubts in industrial applications. In 2002, Theofanous et al. [31] made an impression for the balance on the construction/operation of engineering equipment against reliability/safety concerns by conducting boiling crisis called “*burnout*” experiments on fresh and aged surfaces. The CHF values obtained varied from 50% to 140% of the hydrodynamic instability theory proposed by Kutateladze in 1950 [19] and further developed by Zuber in 1958 [20]. In the study by Theofanous et al. [31] nucleation site density increased with the degree of heater aging, and surface “*aging*” was explained by Marcus and Dropkin [32] in 1965 as the surface topography changed with time due to degassing, contamination, and oxidation. They observed a uniform heat flux after 50 hours of boiling operation in water, where HTC initially decreased but subsequently increased to the value followed by a constant performance for 36 h. Joudi and James [33] analyzed that secondary run-in water augmented the surface bubble population decreasing surface temperature, while Tu et al. [34] noticed an increase in CHF values on freshly sandblasted copper and stainless-steel heaters in deionized (DI). This was attributed to a couple of “*doughnut*” shaped spots appeared at the base of boiling bubbles and became more apparent with prolonged and more vigorous boiling. In the research of Moze et al. [35], one plain and one laser-textured surface by was used. Repeated formation of a vapor film accompanying elevated temperatures, instant low-temperature annealing, can induce chemical and morphological

changes on both surfaces. They analyzed that cuprous oxide (Cu_2O) and cupric oxide (CuO) components resulted at the repeated CHF points. Contrary to the initial performance loss after the first run, the maximum HTC remained steady with the advantage of lower surface temperatures at the additional runs. In another study, Moze et al. [36] aimed to explore the aging behavior on copper surfaces under approximately 40 - 50 h of intermittent boiling operation for eight days in 2020. The HTC on the exposed plain surface exhibited a 21% increase on the second day (3rd run), and this performance remained constant for the rest of the days, while there was no significant change observed on the treated surface. Elkholy and Kempers [37] conducted a sequence of CHF runs on two identical copper surfaces, and HTC performances of the aged identical surfaces increased by up to 22% lower wall superheat. A critical inference was that 25 h pre-boiling was required to obtain repeatable results within a 5% deviation for the same copper surfaces. Similar to the study managed by Joudi and James [33], boiling left many spots pointing out the development of the oxide layer and number of nucleation sites. Interestingly, the latter measurements on surface roughness indicated an increase by an average of 15%. Moreover, Wang et al. [38] experienced surface oxidation on copper and nickel surfaces held in boiling water at saturated conditions for different periods of time (30 min, 7 h, and 24 h) before conducting the pool boiling experiments. It has to be mentioned that the time for the pre-heating process for boiling experimentation has not been taken into account. Nevertheless, both surfaces showed gradually increasing CHF and decreasing superheat values for the same heat fluxes as the operation time increased, while the authors pointed out the effect of surface roughness on wettability by Cu_2O and NiO formation. Furthermore, Min et al. [39] evaluated the long-term wetting and corrosion characteristics on aluminum and copper surfaces treated with water at 82 and 100°C. The corroded surfaces soaked by heated water achieved decent wettability supported by retaining water and a low receding contact angle.

Besides the fact that oxidation has such a large effect on transition and alkali metals, it's believed that mechanical surface treatments show better consistency for long-term applications in line with the needs of the industry. In addition, surface modifications realized utilizing dip coating or chemical deposition techniques, for example, cannot be cost-effectively applied on large scales and show limited compatibility with the surface material and the working fluid, respectively. To improve nucleate pool boiling performance, structured surfaces manufactured through micro-machining, laser-texturing, and 3-D printing can be adopted. By using one of those techniques, micro-channels or micro-holes can be used in order to alter boiling heat transfer (BHT) with systematically varied geometric sizes. Particularly the density of micro-cavities can have a governing influence since the number of active nucleation sites has been reported to be one major affecting parameter for heat transfer. Besides, micro-fins or channels have presented the advantages of induced capillary flow, separating the paths of vapor release and replenishment liquid for many years. It is important to note that the micro-fin or channel structure, width, height, and spacing must be properly configured and optimized to take full advantage of these added benefits, where numerous experiments have been done under several operating conditions. In 1980, Nakajima et al. [40] developed tunnel surfaces enabling vapor escape by tiny pores in water, R-11, and liquid nitrogen, while achieving an 80 - 90% reduction in wall superheat. This detailed research demonstrated the importance of liquid suction, which has been reported to be a driving force for the

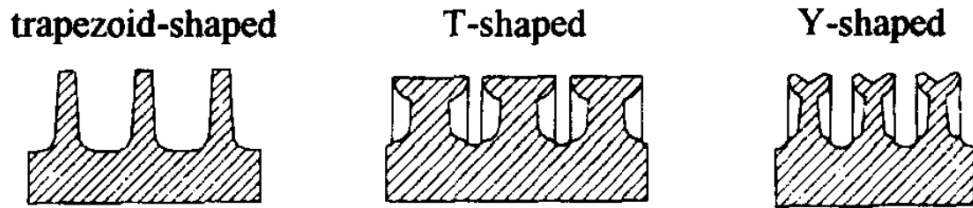


Figure 6: Particular microchannel geometries providing high rate of heat dissipation [44].

bubble formation cycle [41]. Moreover, Arik et al. [42] applied a diamond-base microporous coating on silicon heaters and studied with a dielectric liquid of FC-72 at large pressure and subcooling ranges, where 60% enhancement was achieved over treated surfaces. Then, Memory et al. [43] in 1995, Hubner and Kunstler [44] in 1997 tested commercial re-entrant cavity tubes from the GEWA series (trapezoid-shaped, T-shaped, and Y-shaped) as shown in Figure 6, as well as Thermoexcel and Turbo tubes and achieved up to 20 times higher HTC values at fairly low heat flux conditions. The studies indicated that as the heat flux increases, the performance of these re-entrant cavity tubes reduces significantly. In the same years, Mudawar and Anderson [45] investigated several surface structures of smooth, sanded, rectangular fins, and square studs in FC-72, where micro-structures decreased surface temperature and yielded CHF values in excess of 50 W/cm^2 , three times a smooth surface. In addition, Cooke and Kandlikar [46] used a deep reactive etching method to fabricate the microchannels. The geometrical trends illustrated that wider and deeper channels with thinner finned surfaces showed the best heat transfer methods for water. Kandlikar [46] proposed a hypothesis expressing that the resulting rapid interface between vapor and liquid by evaporation momentum force increases the micro-convection heat transfer around the nucleating bubbles. Then, the developed features of microchannels based on the hypothesis enhanced the heat transfer coefficient eightfold and CHF by two-and-half times. Rajulu et al. [47] analyzed the heat transfer coefficient of the re-entrant cavities in acetone, isopropanol, ethanol, and water between $1.1 - 4.2 \text{ W/cm}^2$, where re-entrant surfaces have a considerable enhancement. Klausner and McKenna [48] utilized a parametric study by alternating the cap size on fin tips and observed the hydrodynamic communication effect on increasing nucleate boiling. On a cylindrical heating surface, Mehta and Kandlikar [49] applied circumferential V-groove microchannels and axial rectangular microchannels and reported that rectangular microchannels outperformed the V-groove. In addition

to basic structures on open microchannels, Walunj and Sathyabhama [50] discussed channel geometries with rectangular, parabolic, and stepped forms, where the channel width and height varied from $250\ \mu\text{m}$ to $500\ \mu\text{m}$ and $500\ \mu\text{m}$ to $800\ \mu\text{m}$, respectively (see in Figure 7). Modifying the rectangular channel into a parabolic and stepped microchannel resulted in a maximum enhancement of 88% and 169% in heat transfer rate. In comparison, the heat transfer coefficient increased gradually in the range of 21.5 - 35.4%.

Overall, the literature has a vast amount of information on segmented or in-line rectangular micro-fins and channels for pool boiling heat transfer. However, possible channel geometries are not discussed in detail in advanced dielectric liquids (FC-72, Novec HFE-7000, and HFE-7100), whereas these chemically inert liquids have low vapor pressure, viscosity, and surface tension [51]. When screening the literature, one can identify that the channel geometries out of reach with traditional production methods may have better performance and can be achievable with additive manufacturing (AM). Specifically, modern laser powder bed fusion (L-PBF) machines can produce fine, complex features such as microchannels [52], lattice, and porous structures [53], which have the potential to enhance a heat transfer coefficient up to 0.7 and 1.8 times. However, some limitations exist as micro-structures become smaller, which are rounded edges instead sharp geometries and surface roughness disadvantages due

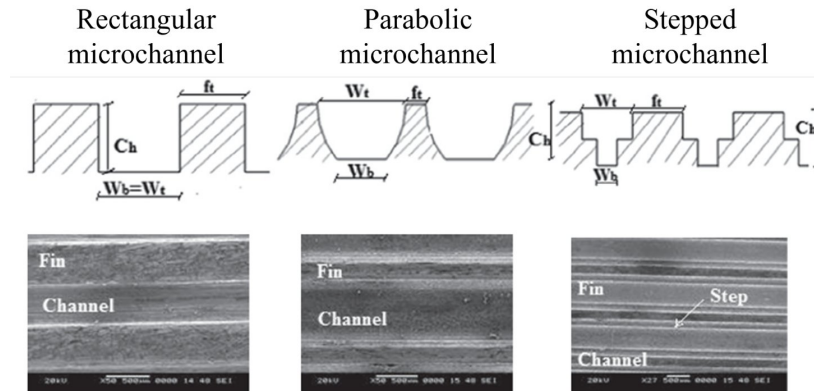


Figure 7: Distinctive channel geometries to control bubble growth [50].

to partially melted or sintered particles [54]. In addition, while the density, size, and depth of the micro-drills have been investigated for different subcooling levels, there is a limited information about the pressure effect combined with micro-drills. In final, once the literature is investigated in detail, it is evident that aging and burn-out conditions considerably affect the BHT of plain metallic surfaces. In order to understand the effect of boiling crisis on test surfaces, a sequence of boiling tests was conducted in [31, 33, 35, 36, 37]. However, apparently, large disagreements can be found in the heat transfer coefficients reported, and there are unclear dependencies for enhancements or gradual decreases of CHF values. In common, the objectives were to understand the aging effect on BHT entitled of “*repeatability on different surface morphologies*”, “*aging in saturated water*”, “*oxidation effect on heating surfaces*”, etc., whereas a few significant parameters and techniques for the aging induced by boiling are still missing. Recently, the boiling crisis with repeating experiments [35, 37] and the aging effect might have been explained by continuing the boiling heat transfer between CHF runs [36], but the change of surface performance induced 24 h continuous boiling at different heat fluxes were not discussed. In addition, the effect of several repeating boiling crises, peroxidation, and surface roughness on long-term boiling performance was not studied in detail yet. In addition, the effect of several repeating boiling crises, peroxidation, and surface roughness on long-term boiling performance was not studied extensively until now. I am running a few minutes late; my previous meeting is running over.

In this study, the objectives are to enhance the BHT with artificial cavities by microdrill manufacturing and additive manufactured microchannel surfaces in addition to understand the oxide layer development and their impact on thermal performance. In the study investigating artificial cavities, smooth reference surfaces added to two surfaces with micro-drills were evaluated under a pressure range of 1 - 1.5 bar and at different subcooling temperatures from 0 to 10 K in DI water. The size of micro-drills

was adjusted to 200 μm and 700 μm , while they were organized in a hexagonal array with a drill-to-drill distance of 1 mm.

Furthermore, the boiling performances of three channel geometries fabricated by laser powder bed fusion (L-PBF) on aluminum substrates: rectangular, V-shape, and inverse V-shape were studied. The roughness effect of the plain AM surface was analyzed and compared with a conventionally manufactured mirror-like polished surface. The measurements were carried out two times for surfaces in saturated Novec HFE-7100 dielectric liquid at atmospheric pressure. Furthermore, the experiments focused on oxide layer development and its effect on heat transfer were carried out on pure copper substrates and tested for various operating scenarios. The experiments were conducted in an in-house developed, fully-automated boiling setup filled with saturated and subcooled dielectric liquids (DI water and HFE-7100) at atmospheric pressure.

Long-term boiling operations were utilized for 24 h with constant heat fluxes, while the effect of alternating series of heat loads on the boiling process was also investigated. For the abovementioned experimental objectives, the prepared surfaces were analyzed by using Contact Angle Measurements, Confocal Microscopy, Scanning Electron Microscopy, and X-Ray Diffraction in order to interpret the surface wettability, the formation of oxide layers, and their effects on overall heat transfer phenomena. For some tests, bubble formation was captured using high-speed imaging techniques.

CHAPTER II

EXPERIMENTAL SYSTEM

This chapter provides a thorough presentation of the experimental system and the measurement techniques employed, offering comprehensive details. The experimental procedure, aligned with the objectives of the study, is elaborately demonstrated. Moreover, a meticulous uncertainty analysis is conducted, considering various factors that significantly impact the measurements. This detailed analysis holds great potential to enhance the accuracy and reliability of the measurements.

2.1 Experimental Setup

The experimental setup consists of several electronic devices and additional equipment according to conditional requirements. The process flow diagram and the close-up view of the test setup have been presented separately in Figure 8 and Figure 9. Besides, the real view at close range and the cross-section of the boiling chamber is provided in Figure 10. The setup was operated and monitored via an in-house developed LabVIEW[®] automation program (see the front panel in Figure 11), while a fully automatic data acquisition was carried out.

The boiling chamber, illustrated in Figure 10a, is constructed from stainless steel and has a 163 mm inner diameter and a height of 225 mm. During the tests, the chamber was filled with either di-ionized water or a dielectric liquid of HFE-7100 by 3M (Minnesota Mining and Manufacturing) Company, occupying a height of 180 mm. Copper substrates (7) were mounted onto a copper heater block (1). The heater block was powered by four cartridge heaters (2) capable of generating a total heat load of 1500 W. To enhance heat transfer between the heater substrate and the heater block, a high-temperature thermal compound with a thermal conductivity of

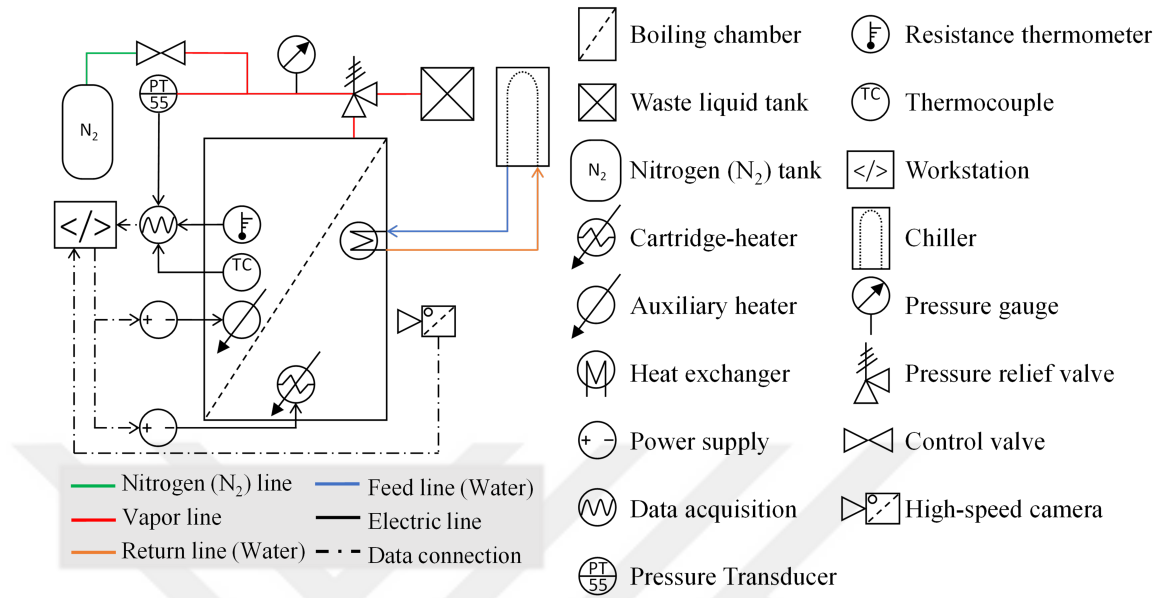


Figure 8: Process flow diagram (PFD) of the experimental setup for boiling tests.

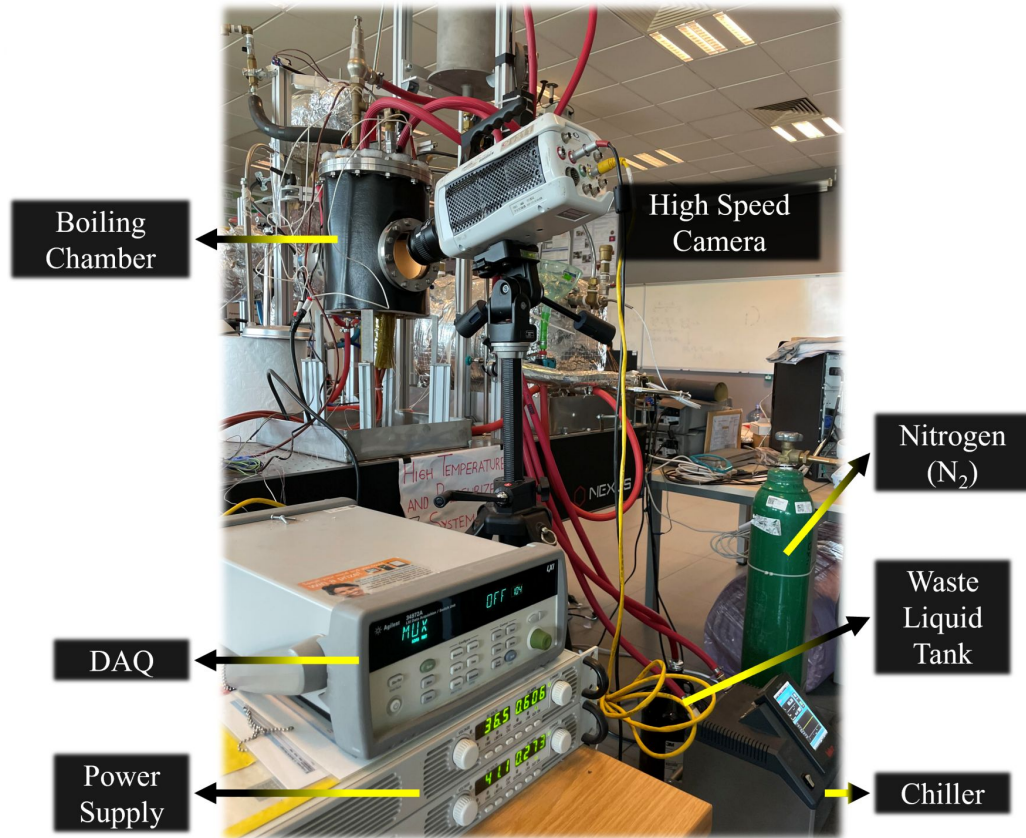


Figure 9: Close-up view of the experimental setup.

13.5 W/mK was applied. The heater substrates were concentrically placed within a conic polyether ether ketone (PEEK) insulator (10), and a Viton O-ring (9) was inserted to prevent any liquid or vapor leakage at the junction point. Additionally, a high-temperature resistance liquid washer was used to eliminate the possibility of satellite bubble activation at the mounting surface. In the system, the heat generation of the cartridge heaters was regulated by an integrated Keysight DC high-power supply (N5750 A), while J-type thermocouples were employed in the cartridge heaters to prevent possible overheating. Two auxiliary heaters (3), powered by a Keysight high-power supply (N5771 A), were immersed in the bulk liquid, with a copper coil heat exchanger (6) positioned 60 mm above the heating surface and connected to a chiller (Huber Ministat125) that circulated cooling water. The auxiliary heaters could provide up to 330 W of heat with feedback temperature control using a resistance thermometer (PT-100) (4), positioned closely to the boiling surface without causing any disturbances to the detaching bubbles. This setup ensured that the liquid remained at the desired temperature and facilitated the condensation of vapor generated by the heated surfaces and auxiliary heaters. The outer surface of the chamber was insulated using a low-conductive material (Polyurethane) to minimize heat losses and maintain temperature stability within the bulk liquid. To maintain the desired pressure within the system and prevent any potential explosions or cracks, highly sensitive pressure relief valve was employed. On the other hand, to increase the system pressure and maintain steady levels, a highly pressurized Nitrogen (N_2) tank reaching pressures up to 125 bars was integrated into the setup. The chamber pressure was measured using a precision pressure transducer (Mesens MPS) with an uncertainty of ± 0.01 bar. Pressure and temperature readings from the operating system were collected using a data acquisition device (Agilent 34872A). To ensure the safety of the heated surfaces and prevent any damage caused by undesired Critical Heat Flux (CHF) conditions, an automated shutdown procedure was implemented.

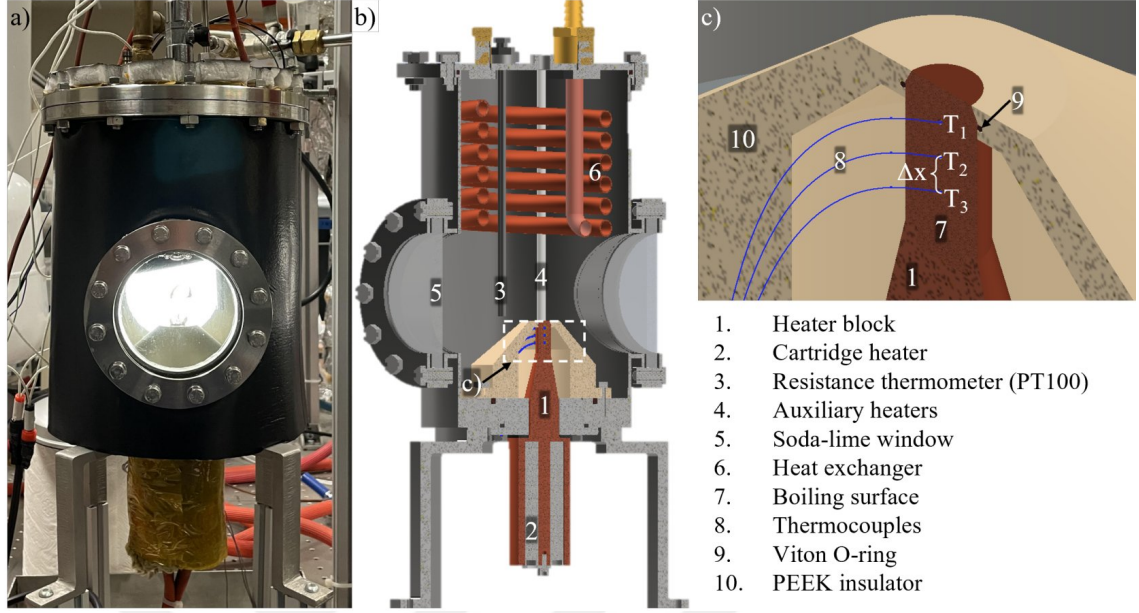


Figure 10: Boiling chamber: Close-up view (a), section view (b), heater block including thermocouples (c).

This procedure would activate when rapid temperature increases of at least 3 K/sec and 0.5 K/sec were detected in the operating sample for DI water and HFE-7100 dielectric liquid, respectively. Bubble growth and departure were captured using a high-speed camera (Phantom V311) operating at a frequency range from 300 to 2000 Hz, with a resolution of $35 \mu\text{m}/\text{pixel}$. The camera was focused through a solid-lime glass (5), and a light source was positioned on the opposite side to facilitate the use of the shadowgraph technique for high-speed imaging.

2.2 Measurement Methods

In this work, the so-called cascaded thermocouples method was used to determine the surface temperature, and hence the heat flux dissipated from the boiling surface. For this method, the signals of three calibrated thermocouples (8) with a diameter of 0.6 mm (see in Figure 10c) were employed. The thermocouples were inserted to the specified in-line locations on the samples by soldering (solder thermal conductivity is 65 W/mK) or using the abovementioned conductive thermal paste. For heat flux

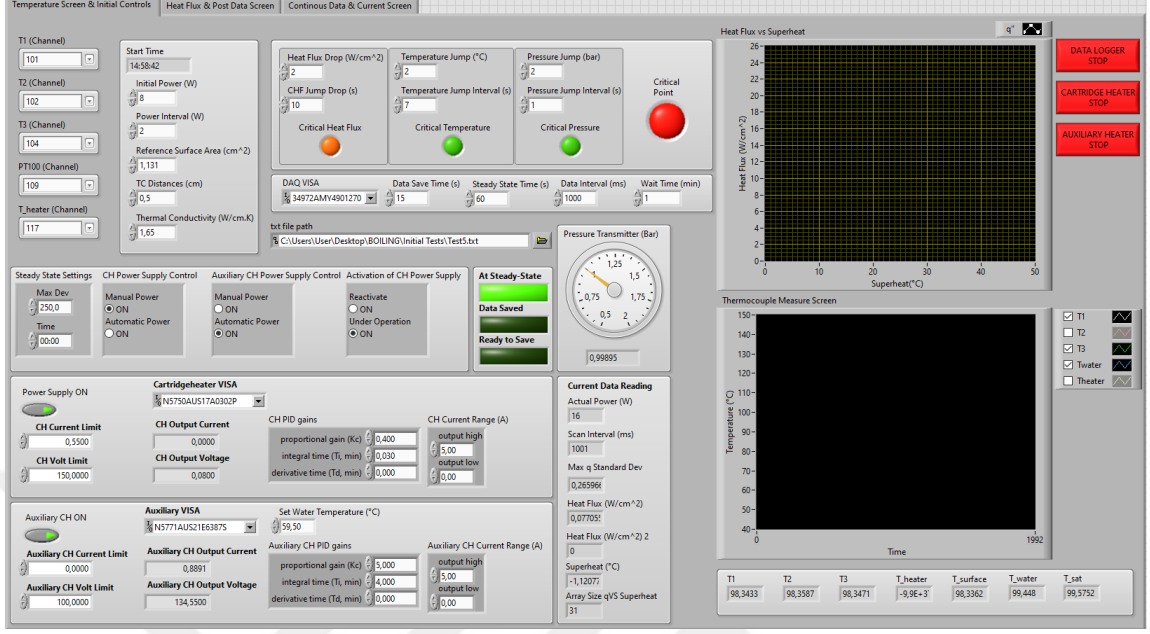


Figure 11: The graphical user interface of the in-house developed LabVIEW® automation program.

computations on the boiling surface, Fourier's law of one-dimensional heat conduction Eq. 10 was utilized as follows:

$$q'' = -k \frac{dT}{dx} \quad (10)$$

where the thermal conductivity of the 99.5% pure copper substrate k_{Cu} were considered as 395 W/mK whereas this value was 170 W/mK and 165 W/mK for the thermal conductivity of the cast aluminum alloy samples k_{Al} and additive manufactured aluminum specimens, respectively [55]. The temperature gradient along the vertical direction towards the copper surface was expressed by Eq. 11 based on the measured temperatures at three different locations:

$$\frac{dT}{dx} = \frac{3T_1 - 4T_2 + T_3}{2\Delta x} \quad (11)$$

T_1 , T_2 , and T_3 are the wall temperature values along the heat flow direction, and Δx is the distance between the thermocouples (5 mm) as shown in Figure 10c.

Considering one-dimensional heat conduction in copper substrates, wall superheat $\Delta T_{superheat}$ is often approximated by Eq. 12 with calculated heat flux unless there is a thermometer measuring surface temperature. In addition, T_1 closely measured at the boiling surface and T_∞ of the bulk liquid, k of the substrate, and the distance Δx_w of the thermocouple to the surface are necessary measures for the following approximation:

$$\Delta T_{superheat} = T_w - T_\infty = T_1 - q'' \left(\frac{\Delta x_w}{k} \right) - T_\infty \quad (12)$$

Finally, the heat transfer coefficient HTC is estimated via Eq. 13 by dividing the calculated heat flux to the surface superheat as:

$$HTC = q'' / \Delta T_{superheat} \quad (13)$$

2.3 Uncertainty Analysis

Moze et al. [56] have observed that the critical heat flux values recorded under similar operation conditions and on similar surfaces often show a significant discrepancy. A detailed uncertainty analysis has been performed, and results are presented in Table 1. The uncertainty of both thermocouples is assumed to be the same; the maximum uncertainty in heat flux through Eq. 14 was found as calculated:

$$u(q'') = \sqrt{2 \frac{k}{\Delta x} u^2(T) + \left(-\frac{(T_3 - T_1)}{(\Delta x)^2} \right)^2 u^2(\Delta x) + \left(\frac{(T_3 - T_1)}{\Delta x} \right)^2 u^2(k)} \quad (14)$$

where the uncertainty in the temperature readings $u(T)$ was calculated as 0.15 K and is expressed as follows:

$$u(T) = \sqrt{u_{cal}^2 + u_{cbath}^2} \quad (15)$$

with the uncertainties in the calibration u_{cal} and calibration bath temperature u_{cbath} are ± 0.15 K and ± 0.03 K. Besides, the uncertainty of the thermal conductivity

of substrate material was specified as ± 7.9 and 4.7 W/mK for the specimen materials of copper and aluminum, respectively. Finally, to validate the measurement of heat flux (justification of the assumption of one-dimensional heat conduction assumption), the linearity of temperature gradients between thermocouple readings was tested at different heat fluxes [57] and a good agreement was obtained as shown in Figure 12.

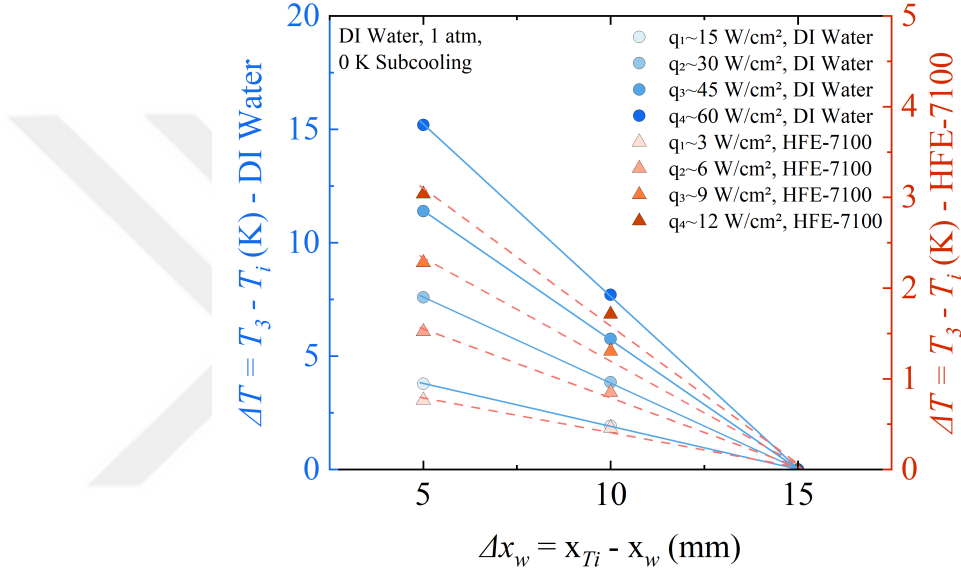


Figure 12: The temperature profile of the thermocouple measures during a sample CHF run process in DI water and HFE-7100 for heat flux validation.

The following expression for the uncertainty calculation of wall superheat is used:

$$u(\Delta T_{superheat}) = \sqrt{u^2(T_1) + u^2(T_\infty) + \left(-\frac{\Delta x_w}{k}\right)^2 u^2(q'') + \left(\frac{q'' \Delta x_w}{k^2}\right)^2 u^2(k) + \left(-\frac{q''}{k}\right)^2 u^2(\Delta x_w)} \quad (16)$$

where the uncertainty of the PT-100 thermometer measuring bulk liquid temperature is given as:

$$u(T_\infty) = \sqrt{u_{cal}^2 + u_{conv}^2} \quad (17)$$

It is found to be ± 0.41 K. The uncertainty of the thermocouple location relative to another thermocouple $u(\Delta x)$ and the surface $u(\Delta x_w)$ is expressed for the uniform heat flux measurements separately:

$$u(\Delta x) = \sqrt{2 \left(-\frac{u_{hole}}{\sqrt{3}} \right)^2 + 2 \left(-\frac{u_{TC}}{\sqrt{3}} \right)^2} \quad (18)$$

$$u(\Delta x_w) = \sqrt{2 \left(-\frac{u_{hole}}{\sqrt{3}} \right)^2 + 2 \left(-\frac{u_{TC}}{\sqrt{3}} \right)^2} \quad (19)$$

where the uncertainty of u_{hole} and u_{TC} are the uncertainty of the thermocouple hole position on the specimen and thermocouple position in these openings. Finally, the relative uncertainty $\Delta u(HTC)$ of the heat transfer coefficient was calculated by the following equation:

$$\Delta u(HTC) = \sqrt{\left(\frac{u(q'')}{q''} \right)^2 + \left(\frac{u(\Delta T_{superheat})}{\Delta T_{superheat}} \right)^2} \quad (20)$$

Table 1: Experimental uncertainties for measured and derived parameters.

Parameter	Uncertainty	
	Solder	Thermal Paste
Temperature reading - $u(T)$, $u(T_w)$ [K]	± 0.15	± 0.15
Bulk liquid temperature - $u(T_{inf})$ [K]	± 0.41	± 0.41
Calibration bath temperature - u_{cbath} [K]	± 0.03	± 0.03
Convective temperature difference - u_{conv} [K]	± 0.4	± 0.4
Calibration (Thermocouple, PT-100) - u_{cal} [K]	± 0.15 , ± 0.06	± 0.15 , ± 0.06
Thermocouple position to other one - $u(\Delta x)$ [mm]	± 0.07	± 0.07
Thermocouple position to surface - $u(\Delta x_w)$ [mm]	± 0.05	± 0.05
Thermocouple position in hole - u_{TC} [mm]	± 0.05	± 0.1
Thermocouple hole position - u_{hole} [mm]	± 0.07	± 0.05
Thermal conductivity (Cu, Al) - $u(k)$ [W/cmK]	± 7.9 , 4.7	± 7.9 , 4.7
Superheat - $u(\Delta T_{superheat})$ - max [K]	± 0.41	± 0.13
Heat flux - $u(q'')$ - max [W/cm ²]	± 3.23	± 1.36
Heat transfer coefficient - $\Delta u(HTC)$ - max [%]	± 2.6	± 1.9

Furthermore, the list of detailed uncertainty studies is presented in Table 1 for the two different measurement systems diverged according to installment methods of the inline thermocouples by soldering or using high-conductive thermal paste.

2.4 Experimental Procedures

The experimental procedure employed in this study was varied depending on the specific research objectives. This section provides a summary of the common procedures while highlighting the specific methods utilized in this comprehensive study. The common experimental procedures, visually depicted in Figure 13, are described as follows:

1. In experiments conducted using DI water, after completing thermocouple calibration, any unused substrates were stored in an evacuated desiccator containing silica gel to prevent possible oxidation at room temperature. Conversely, for experiments using HFE-7100 dielectric liquids, thermocouples were primarily mounted on the sample using thermal paste, and only the thermocouples were calibrated. The thermocouple sockets remained connected to the data acquisition (DAQ) device throughout the calibration process. Once the experimental setup was installed and the substrate was mounted, the working fluid was poured into the chamber.
2. The working liquids inside the chamber were subjected to boiling at a temperature higher than their respective saturation temperatures for a duration of 1 hour. The pressurized vapor generated during this process was continuously released through a check valve into a waste liquid tank. This step was implemented to minimize the presence of dissolved non-condensable gases in the system. Subsequently, the degassing valve was closed, and the liquid was gradually cooled down to reach the desired temperature conditions, and the pressure naturally decreased to atmospheric levels.

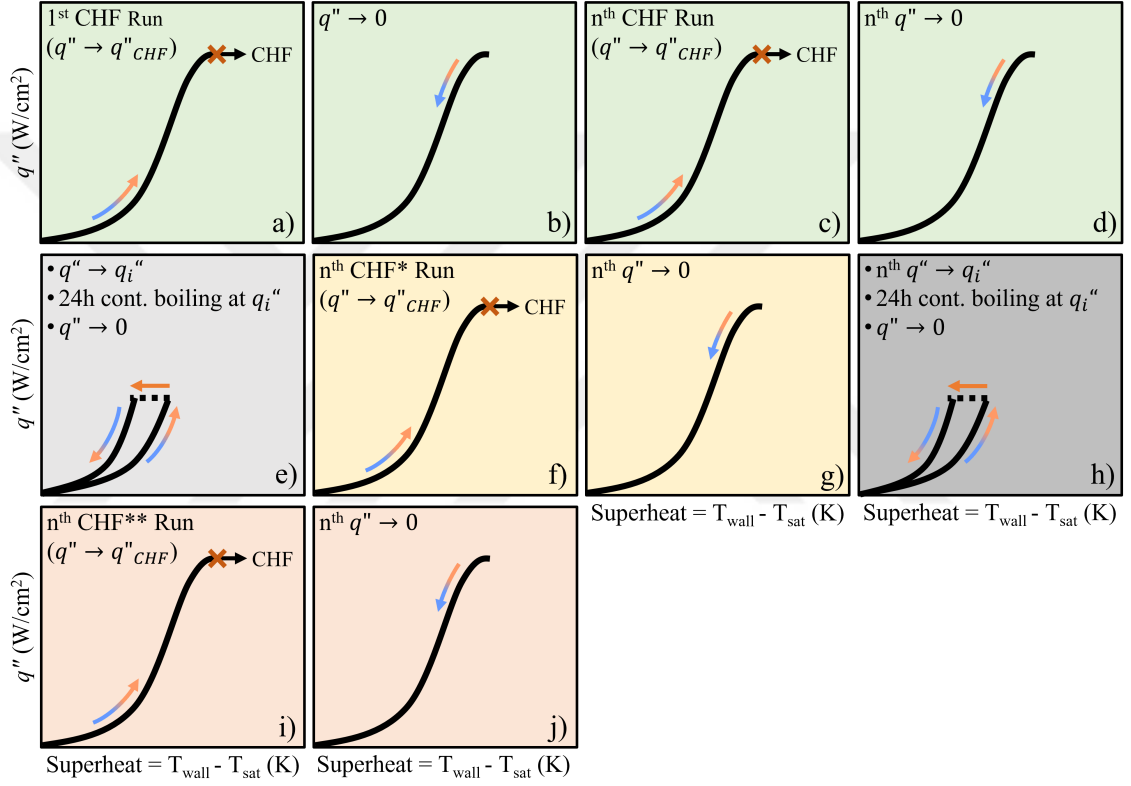


Figure 13: Experimental procedure for alternating heat removal tests: Heating for CHF run (a), cooling procedure of the surface down to saturation temperature (b), heating for CHF* run (c), surface cooling procedure (d); 24h continuous boiling operation at constant heat flux level (e), heating for CHF* experiment (f), surface cooling procedure (g); secondary continuous boiling operation at constant heat flux (h); heating for CHF** run (i), surface cooling procedure (j).

3. In order to simulate an alternating heat removal operation, like in real applications, the surface was carefully heated up with a heat flow rate of 1 W/min until a boiling crisis ($q'' \rightarrow q''_{CHF}$) occurred, i.e. CHF due to vapor film formation bringing a drastic reduction in heat transfer existing over the heated surface as well as rapid rise in surface temperature, where this is identified as 1st CHF run (see Figure 13a).

4. Afterwards, the surface was cooled down to the temperature of the working liquid ($q'' \rightarrow 0$) (see Figure 13b). As needed, subsequent CHF runs were executed immediately with the same heat flow rate to achieve maximum heat fluxes (see Figure 13c) under the alternated or same conditions. Once the surface reached CHF, the surface temperature was again reduced to the desired temperature of the liquid (see Figure 13d).

5. In scenarios where an aging process is required for the operating specimens, the subsequent step involved heating the surfaces to predefined heat flux levels of 15, 30, 45, and 60 W/cm² for DI water, and 6 and 12 W/cm² for HFE-7100 dielectric liquid. These heat flux levels were deliberately set lower than the previously measured Critical Heat Flux (CHF) values (see in Figure 14). The surfaces were then subjected to continuous boiling operation for a duration of 24 hours (as shown in Figure 13e). Following this procedure, the surfaces were cooled down, and the experimental procedures could be carried out by either performing a final heat-up to CHF points (referred to as "CHF* runs") to observe the surface performance after the aging process, or continuing with subsequent CHF* runs (as illustrated in Figure 13f-g).

6. In one of the scenarios, an extended exposure to continuous boiling performance was necessary, and a secondary 24 hour continuous boiling operation was carried out (see in Figure 13h). Finally, a heat-up process with the same heat flow rate was

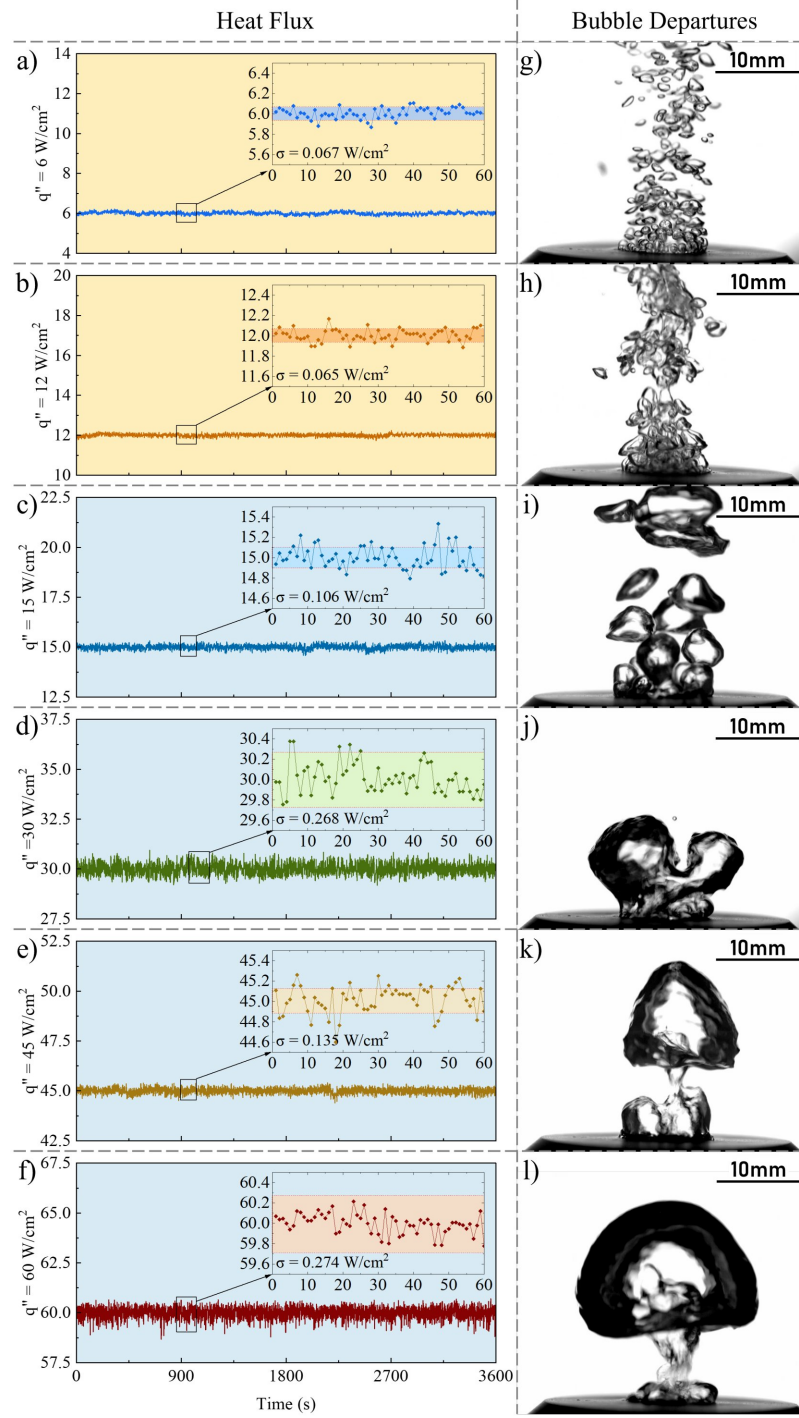


Figure 14: Sequences showing the temporal variation of heat fluxes applied for 24 hour during long-term operation and corresponding exemplary images of bubble departures at 6 W/cm^2 (a-g) and 12 W/cm^2 (b-h) for HFE-7100 (yellow painted); 15 W/cm^2 (c-i), 30 W/cm^2 (d-j), 45 W/cm^2 (e-k), and 60 W/cm^2 (f-l) for DI Water (blue painted).

performed to reach the maximum heat flux condition (see in Figure 13i), followed by decreasing the surface temperature by turning off the input power (see in Figure 13j). Alternatively, in some cases, a specific Critical Heat Flux (CHF) run may not be required, and the scenario can be completed by solely utilizing the guidance provided in the 5th one presented in Figure 13e.

During 24 h long-term operations, the constant application of heat flux is important and had to be ensured in order to justify similar test conditions. For this purpose, the temporal variation of heat fluxes is shown in Figure 14 as randomly chosen sequences over 3600 sec. for 6 and 12 W/cm² in HFE-7100, and 15, 30, 45, and 60 W/cm² in DI water. It can be seen that for all heat flux levels, the standard deviation resulted below 0.3 W/cm², while relatively, the largest fluctuation resulted for 30 W/cm² heat flux. The largest standard deviation was found for 60 W/cm² heat load; however, this represents a relatively low fluctuation of approximately 0.46%.

2.5 Surface Preparation

The heating substrates were manufactured with 12 mm outer diameter and 25 mm height as depicted in Figure 15k. Copper and aluminum were preferred as test material in the current study to imitate the configuration of thermal management system for electronic cooling such as copper electronic chips, mini heat exchangers, conductors etc. Another objective of the used materials is to provide a rather homogeneous temperature distribution over the boiling surface with 10 mm diameter. The Figure 16 visualizes the preparation process of the specimens to be experimented corresponding to thermocouple insertion method onto specimens. In general (I), after the manufacturing process, the heater surfaces possessed micro-scratches and reentrants which were step-by-step smoothed by using silicon carbide abrasive papers starting from #180 up to #7000 grits. Then, a water-based suspension containing 1 μ m monocrystalline diamond particles (Metkon DIAPAT-M) were used with 1 μ m

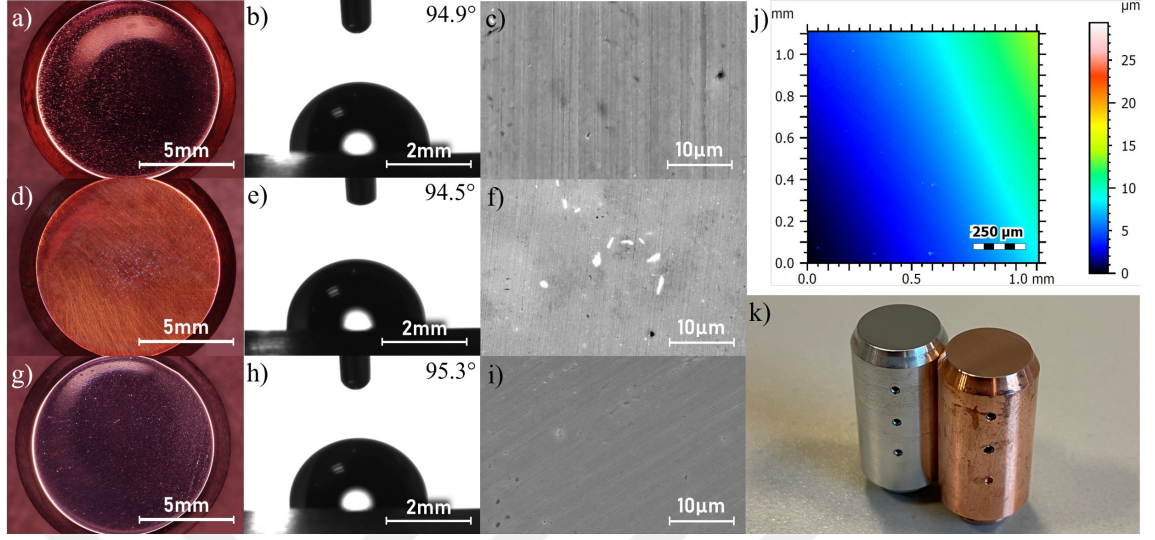


Figure 15: Microscopic images, contact angle measurements, scanning electron microscope images of the polished copper surface before the calibration (a, b, c), the polished copper surface after the calibration (d, e, f), the polished aluminum surface (g, h, i); surface roughness analysis of the experimental surfaces (j); real illustration of the sample fabricated specimens (k).

polishing cloths (FEDO-1S) to reduce the size of scratches and to get a well-defined mirror-like finished surface condition. An average surface roughness $R_a = 0.07 \mu\text{m}$ was achieved on all surfaces in general (see Figure 14j).

In the calibration process executed for the experiments, three T-type thermocouples were soldered into each heater specimen and connected to a data acquisition system (DAQ). Subsequently, a calibration was conducted between 5 and 165°C for 7 hours in a water bath. The recordings have been calculated, and the equations resulting in linear interpolation were entered into the system for each thermocouple. A minor pre-oxidation was observed on the specimen surfaces during this phase; thus, a couple of static contact angle measurements were utilized with the sessile droplet method on a Tensiometer (Theta Lite Optical) as illustrated in Figure 17. The samples were positioned so that the surface was horizontally aligned, and the droplet size at the needle was kept constant at $10 \pm 0.1 \mu\text{L}$ and $4 \pm 0.1 \mu\text{L}$ for DI water and HFE-7100, respectively. The average measured values were $94.9 \pm 4^\circ$ (see Figure 15e)

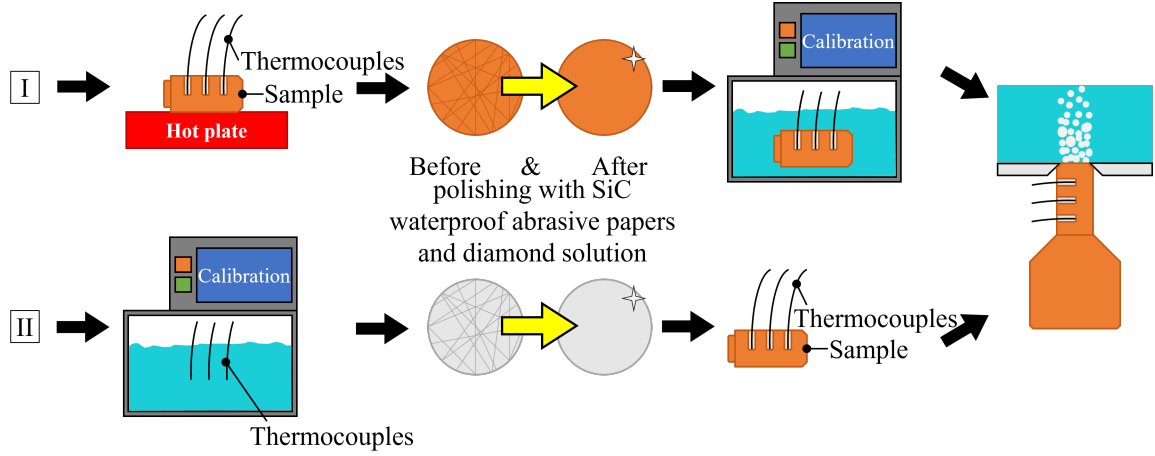


Figure 16: Preparation of the specimens to be experimented, which are soldered to the thermocouples (I) and attached with a high conductive thermal paste (II): Insertion of thermocouples into measuring positions in the sample, surface polishing with silicon carbide (SiC) sandpaper and water-based suspension containing $1\ \mu\text{m}$ monocrystalline diamond particles, calibration in thermal bath between 5 and 165°C , placement of the specimen onto the heating block.

and $23.4 \pm 3^\circ$ on the polished surface. This analysis demonstrated that the calibration process did not affect the CA. In other words, the surface wettability behavior, despite visible tarnish on the boiling surfaces, appeared to be quite stable.

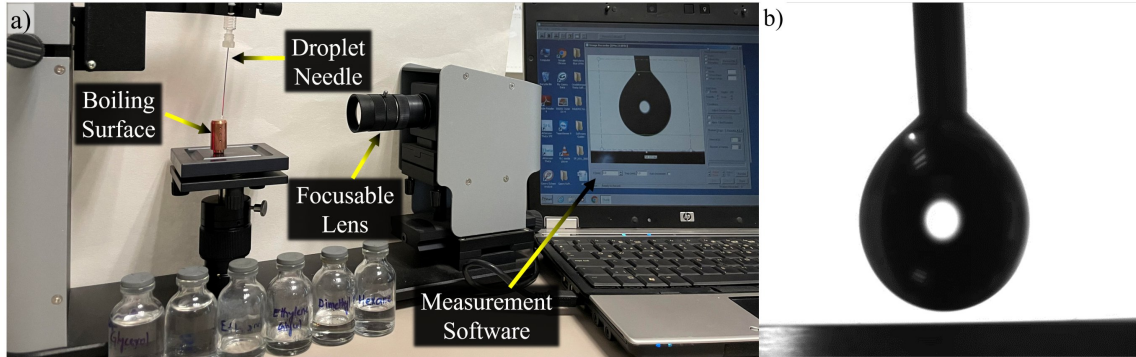


Figure 17: Contact angle measurement (a) and single sessile droplet (b).

CHAPTER III

EXPERIMENTAL RESULTS

In this chapter, the results of the experiments executed for different objectives were detailedly discussed in separate sections: Oxidation Effect on Boiling Heat Transfer, Boiling Heat Transfer Enhancement on Micro-drilled Surfaces, and Boiling Heat Transfer Enhancement on Additive Manufactured Micro-channel. In this section, distinctive production, operation, and analysis techniques were utilized and presented for the abovementioned research objectives.

3.1 Oxidation Effect on Boiling Heat Transfer

Table 2 provides a comprehensive summary of the operating conditions employed in this study. A total of 13 heater specimens made of pure copper were manufactured and tested. The measurements were divided into four distinct categories, each targeting specific aspects of the investigation, as indicated in the first column of Table 2. Additionally, two of the heaters underwent surface treatment using silicon carbide abrasive paper to achieve a specific surface roughness, allowing for the examination of its influence on boiling heat transfer (BHT). The experimental study consisted of four categories to investigate various aspects of the boiling heat transfer (BHT) phenomenon. The first category focused on ensuring experiment repeatability and examining the effect of heat flux load over a 24 hour period, followed by a final critical heat flux (CHF) run to assess any changes in thermal performance. The second category explored the influence of the number of subsequent CHF runs, a 24-hour long-term operation, and a final CHF run. The third category analyzed the effects of long-term operation, including a second 24 hour run and a final CHF measurement. In the fourth category, two heaters were treated with silicon carbide abrasive papers

Table 2: Design of experiments with categories of operation scenarios to observe the oxide layer impact.

Category/ Objective	Surface	Sample no (#)	CHF Run				24 h long-term operation at q_i (W/cm ²)	CHF* Run		24 h long-term operation at q_i (W/cm ²)	CHF** Run
			1 st	2 nd	3 rd	4 th		1 st	2 nd		
Repeatability of CHF measurement and effect of heat flux level during long-term operation	Smooth	1	+	+			15	+			
		2	+	+			30	+			
		3	+	+			45	+			
		4	+	+			60	+			
Effect of number of subsequent CHF runs and long-term operation	Smooth	5					45	+			
		6	+				45	+			
		3	+	+			45	+			
		7	+	+	+		45	+			
Extended alternating long- term operation		8	+	+	+	+	45	+			
	Smooth	9					45				
		10	+	+			45	+	+		
		3	+	+	+		45	+	+		
Effect of surface roughness		11	+	+	+		45	+	+	45	+
	Smooth	3	+	+			45	+			
	#320	12	+	+			45	+			
	#120	13	+	+			45	+			

+ Performed experiment

* After a continuous 24 h long-term operation at q_i''

** After a second continuous 24 h long-term operation at q_i''

⊕ SEM image is available

⊕⊕ SEM image and X-Ray diffraction (XRD) analyses are available

to modify the surface roughness, and similar operating conditions as in the first category were applied, except the 24-hour operation was conducted at 45 W/cm^2 for those heaters. Various techniques, such as contact angle measurements, confocal microscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD) analysis, were utilized to analyze the surfaces of selected heaters.

To ensure the reliability of our experimental data, a comparison of the critical heat flux (CHF) curves obtained from our experiments with those reported in previous studies by Moze et al. (2019) [35], Wang et al. (2019) [58], and You et al. (2016) [59] were conducted. In Figure 18, the CHF curves of 9 heater specimens from our study are presented, alongside the CHF curves from the aforementioned works, which were conducted under similar operational and surface conditions (polished) as our investigation. This comparative analysis serves as a validation of our data and establishes the credibility of our results.

Overall, the data obtained in this study demonstrates a significant similarity among the CHF curves, but there is still a noticeable discrepancy in the range of approximately 70.1 to 84.7 W/cm^2 in terms of CHF values and corresponding wall superheat ranging from 22.5 to 37.5 K . These differences are surely attributed to factors such as the possible uneven distribution of thermal paste between the heater specimens and the heating block, as well as manufacturing tolerances. These effects can have a more pronounced impact on the heat transfer performance of super-polished surfaces, which have nanoscale scratches and require higher surface temperatures to initiate bubble growth. It is important to note that such variations in BHT on similar surfaces with low surface roughness can be unavoided, although they are not extensively reported in most studies. In the subsequent subsections, the measurement results and key findings obtained from different operation scenarios will be discussed.

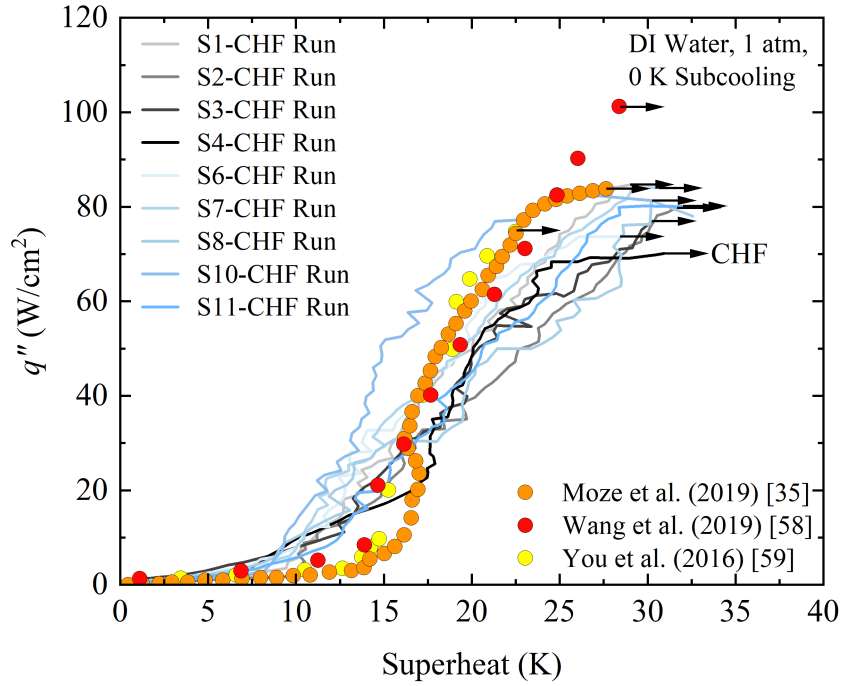


Figure 18: Comparison of boiling curves obtained after initial critical heat flux (CHF) runs on each heater specimen with CHF curves found in the literature (Moze et al. (2019) [35], Wang et al. (2019) [58], You et al. (2016) [59]) for boiling on bare copper surfaces in saturated de-ionized water at atmospheric conditions.

3.1.1 Repeatability of CHF Measurements and Effect of Long-term Operation

In this section, the results of repeating CHF measurements and the impact of heat flux levels during long-term operations are presented. The experimental procedure involved two consecutive CHF runs, followed by a continuous 24-hour boiling operation and another CHF run to observe any changes in CHF. During the 24-hour long-term operation phase, heat flux levels ranging from 15 W/cm² to 60 W/cm² with 15 W/cm² intervals were investigated. Graphs in Figures 19a-b-d-e-g-h-j-k illustrate the typical boiling curves showing heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) as a function of wall superheat ($\Delta T_{superheat}$). Figures 18c-f-i-l demonstrate the temporal variation of HTC during the 24-hour continuous boiling operation, with the color legends indicating changes in wall superheat. When examining the thermal

performance of specimens S1 to S4, the heat flux (q'') after the 2nd CHF run showed a similar trend in the range of approximately 15 W/cm² to 45 W/cm² compared to the 1st CHF run (see Figures 19a-d-g-j). However, beyond $q'' > 45$ W/cm², the 2nd CHF occurred earlier with a lower value of approximately 6% to 13% compared to the 1st CHF. Additionally, the wall superheat changed by approximately 5 K to 8 K between runs. Similar observations were reported by Moze et al. [35], where a 10% decrease was observed after the 2nd CHF run, and subsequent CHF runs did not result in significant changes in CHF values or wall superheat. Consequently, 2nd CHF values remained within the range of 69.5 W/cm² to 73.9 W/cm² at inconsistent wall temperatures for all specimens. The dependency of HTC on repeatability did not exhibit a clear trend. While the maximum HTC values for specimens S1 and S4 decreased after 2nd CHF run, the heat transfer coefficients of S2 and S3 remained relatively stable with similar wall superheat values. The data on the temporal development of HTC during the 24 hours long-term operation revealed an increase in performance ranging from 19% to 29%, accompanied by a decrease in wall superheat of up to 5 K. The highest enhancement of 52% in HTC was achieved during continuous operation at 60 W/cm², indicating a significant dependence of BHT on the applied heat flux level. Particularly for the highest heat flux load, this enhancement became more pronounced after approximately 8 to 9 hours of operation. The long-term operation and changes in wall superheat were clearly reflected as a shift of the boiling curves (obtained after 1st CHF*) towards the left, as observed in Figures 19c-f-i-l. Similar gradual increases in HTC over time and shifts of boiling curves were reported by Moze et al. [36] in their study on bare copper surfaces. Simultaneously, noticeable enhancements of up to 40% were observed in 1st CHF* values after 24-hour continuous boiling operation compared to 2nd CHF values. Correspondingly, heat transfer coefficients ($HTCs$) also showed improvements for all specimens, ranging from approximately 2.6 - 2.85 W/cm²K up to a maximum HTC of 4.13 W/cm²K. These

changes in critical heat fluxes and $HTCs$ can be attributed to several factors, including alterations in surface wetting conditions and the number of active nucleation sites resulting from the thermal insulation caused by the presence of a vapor cushion at the vapor-solid interface.

In boiling heat transfer, evaporation is the primary mechanism for heat dissipation from the surface, while convective and quenching heat contributions are relatively minor. As the heat flux increases, the size of the vapor layer film formed on the heating surface also increases. Simultaneously, the number of departing bubbles and lateral bubble coalescence intensify, leading to the formation of a vapor film layer [58]. This stage is typically the beginning of the critical heat flux phenomenon known as film boiling, characterized by high temperatures ranging from 210 to 320°C. At these elevated temperatures beneath the vapor layer, low-temperature annealing can occur, resulting in the formation of oxide compounds on the heating surface. Macroscopic changes in surface conditions for specimens S1 to S4 can be observed in Figures 20a-b-c-d. Additionally, changes in surface wetting were determined by measuring static contact angles after the 1st CHF* runs, as shown in Figures 20e-f-g-h. An increasing trend in contact angle values was observed from S1 to S4, with specimen S4 exhibiting superhydrophobic behavior with an average contact angle of approximately 118.4°. However, it should be noted that contact angle measurements might not accurately reflect the effect of nano-micro-oxide particles on the overall thermal performance, as the strong surface tension of water prevents penetration into the micro-structures where air pockets are trapped (*Cassie* state [60]). Therefore, contact angle measurements may not be suitable for determining the potential reason behind the improved thermal performance due to the presence of oxide particles at the nano-microscale.

When looking deeper into the surface topography (see Figures 20i-j-k-l-m-n), different types of oxide formations were present. For a constant heat load of 30 W/cm²,

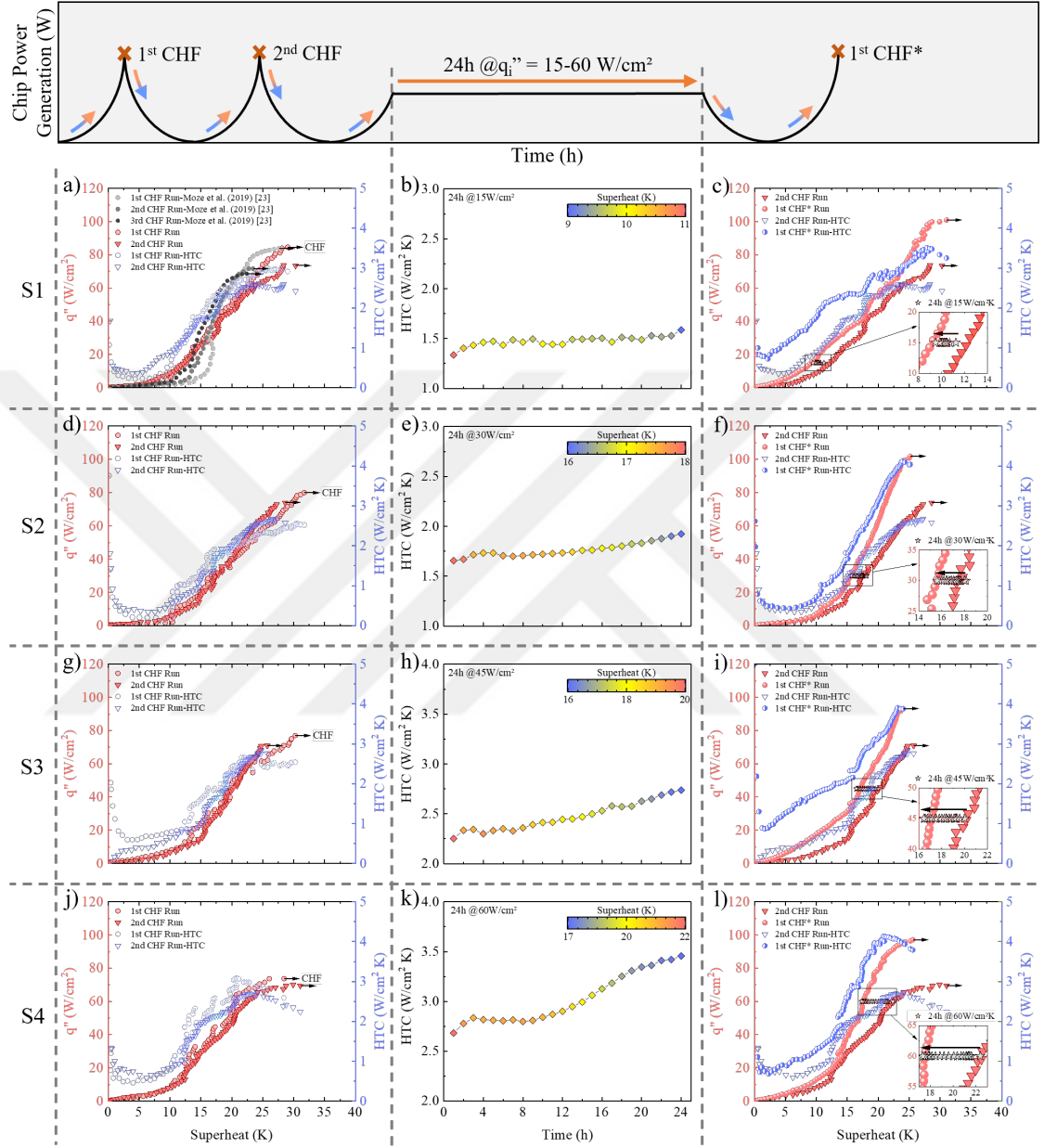


Figure 19: Heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) versus wall superheat obtained after 1st and 2nd CHF run (a, d, g, j) for specimens S1 - S4; temporal change of HTC over 24 h continuous boiling operation (b, e, h, k) conducted at constant heat fluxes of 15 W/cm² (S1), 30 W/cm² (S2), 45 W/cm² (S3), and 60 W/cm² (S4) with wall superheat indication; comparison of heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) measured after 2nd CHF and 1st CHF* run including indication (red arrow and stars) of the change of wall superheat within same duration at corresponding constant heat flux level (c, f, i, l).

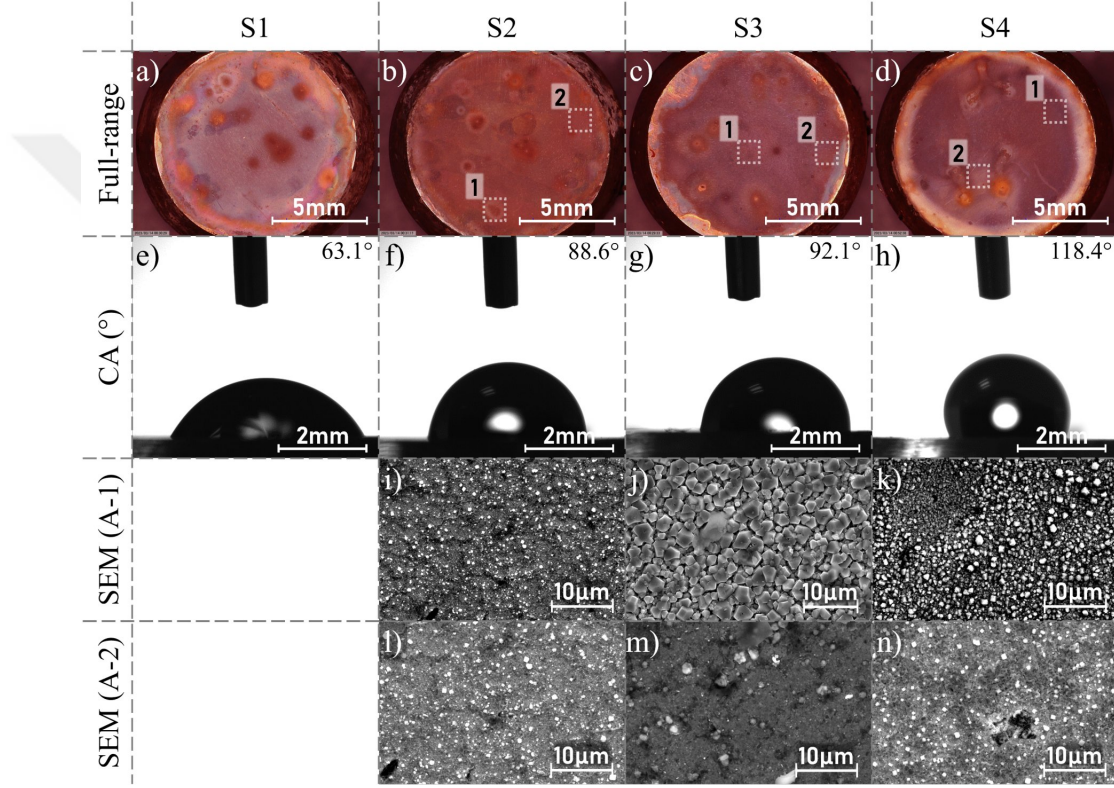


Figure 20: Full range microscopic images with identified areas A-1 and A-2 (b, c, d); average static contact angle (CA) measurements (e, f, g, h); SEM images of the indicated corresponding areas A-1 (i, j, k) and A-2 (l, m, n) on the specimens exposed to boiling experiments 15 W/cm^2 (S1, SEM images are not available), 30 W/cm^2 (S2), 45 W/cm^2 (S3), and 60 W/cm^2 (S4), all measurements were done once each series of experiment was accomplished.

a homogenous cancellous-like oxide layer was formed with arising tiny cubic oxide crystals (see in Figure 20i-l) compared to the reference surface after exposure to boiling in the study by Moze et al. in 2020 [36]. This effect possibly enhanced the active nucleation sites with increasing surface roughness, whereas this parameter was found unchanged as the well-known Wenzel theory [61] was considered [58, 62]. On the contrary, the contact angle was measured on S2, which remained nearly constant at 88.6°C, while this theory worked on the S1 by explaining the enhanced surface roughness with decreasing CA to 63.1°C. A-1 area is the visual evidence of the high deterioration (see Figure 20j), on where large densely packed fish skin-like layer of oxide particles formed, while a porous structure at the microscale was observed in A-2 (see Figure 20m). The particle sizes enlarged up to 10 times of the oxide formations on S2, which might have manipulated the surface roughness with irregularities. The crevices between the oxide formations were maybe able to transport fresh liquid from both directions and enhance the capillary wicking [63]. As given in Kim et al. [58, 64] hypothesis, this could augment the CHF of the S3 upper limits by prolonging the surface rewetting at higher heat fluxes, where the dry-out patches occur at high frequencies, and the probability of vapor film is high. On the other hand, the oxide layer on A-2 proves the non-uniformity of the S3. The heterogeneous surface condition most possibly contributed to the boiling performance with the naturally arisen surface roughness, while the active boiling areas/centers were directly associated [65, 66]. In the final, smaller oxide crystalline particles were observed in a heterogeneous distribution on S4, where the rest of the surface was enveloped by a porous structure like A-2 on S2.

Despite the presence of ambiguous bubble departures on the substrates, which resulted in different surface topographies, the CHF enhancements observed after 24 hours of operation showed unexpected consistency among all specimens. The shift in boiling behavior on the oxidized surfaces can be attributed to the presence of

Table 3: Summary of CHF and HTC s together with performance changes of specimens S1, S2, S3, and S4.

Specimen no. (S#)	Heat flux for 24h operation (W/cm ²)	1 st CHF (W/cm ² K)	2 nd CHF (W/cm ²) (%)	1 st CHF* (W/cm ²) (%)	Change for 24 h operation in HTC (W/cm ² K) (%)	superheat (K)
1	15	o84.7	●73.4 -13%	101.0 38%	0.25 19%	-1.78
2⊕	30	o80.0	●73.9 -8%	101.8 38%	0.27 16%	-2.52
3⊕⊕	45	o77.0	●70.9 -8%	93.2 32%	0.49 22%	-3.43
4⊕	60	o73.7	●69.5 -6%	97.1 40%	0.77 29%	-5.00

Performance change (%) = 1 - Current CHF/Reference CHF (○, ●)

○ Reference value for the CHF performances

● Reference value for the CHF* performances

* After a continuous 24 h long-term operation at q''_i

⊕ SEM image is available

⊕⊕ SEM image and X-Ray diffraction (XRD) analyses are available

microchannels between the oxide particles and the varying orientations of pores within the cancellous structures. The gradual accumulation of oxide particles increased the size of cavities compared to micro-channels on bare surfaces. These larger cavities, which mitigate the inhibitory effects of surface tension forces, allowed bubbles to grow more freely. Additionally, the naturally developed surface topography, including micro-scale reentrants, trapped vapor and facilitated bubble growth at lower wall temperatures during boiling. Furthermore, as the boiling curves were examined at higher heat fluxes, an increase in wall superheats was observed. This increase can be explained by a thinner thermal boundary layer and a narrower range of effective cavity sizes, which promote the activation of previously inactive nucleation sites. On the other hand, elevated surface temperatures result in higher temperatures of the liquid adjacent to the cavities, reducing surface tension and capillary forces. Consequently, the liquid is less likely to be drawn into the cavity, allowing bubbles to grow and detach more easily. This phenomenon is particularly pronounced in the smaller cavities formed by the oxide deposits. Table 3 provides a summary of the CHF values and the corresponding rates of enhancements/reductions compared to the reference values indicated in the grey cells. The yellowish columns in the table present the results of HTC , and wall superheat changes after 24 hours of operation.

3.1.2 Factors influencing oxide particle formation and their impact on thermal performance

As assumed by Moze et al. (2019) [35], we believe that the copper oxide particles covering the surfaces of specimens used in this study comprise cuprous oxide (Cu_2O) and cupric oxide (CuO) components. Thin films of Cu_2O and CuO oxides were developed by direct oxidation of physical vapor deposited copper films in an open atmosphere at between 250 - 400°C by Cruz et al. [67]. They deduced that the grain sizes increase well defined and mostly cubic shapes result as the exposure temperature rises. Moreover, Shariffar et al. [68] used vacuum conditions and Akgul et al.

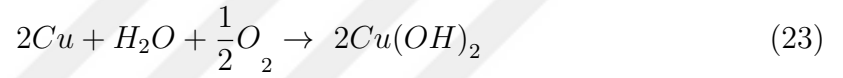
[69] atmospheric pressure, while a similar trend was detected for cubic-shape cupric oxide (CuO) grains on copper films, even when the exposure temperature increased. This could be a strong explanation for the increase of grain sizes on cancellous-like structured specimen S3, while S2 has a smoother surface similar to the surfaces attained in the work of Cruz et al. [67] and Khedir et al. [70]. Figueiredo et al. [71] showed mixed Cu-Cu₂O smooth formation comprising cubical shaped tiny grains by annealing copper foils at 100°C, whereas those at 200 and 300°C showed a single cubic Cu₂O formation. A different method of etching in ammonia was utilized on copper substrates by Liu et al. [72]. A superhydrophobic catkin-like structure was observed similar to the prepared copper surfaces with a hot water treatment at about 75°C by Hassan et al. [73]. Moreover, Saadi et al. [74] demonstrated a wide variety of metal intercepting oxide components resulting in hot water at 75 - 95°C. They reported knife-like copper oxide formations after 18 h of treatment. As the abovementioned studies, under normal atmospheric conditions, knife-like/leaf-like cupric oxide formations (CuO) are thermodynamically more prone to form as the primary oxide layer as follows [75]:



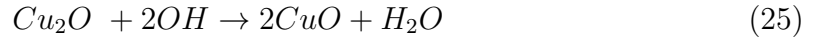
In the meantime, heat, temperature, and time can trigger a partial conversion of copper oxides from one form to another. Copper oxides, such as cuprous oxide (Cu₂O) and cupric oxide (CuO), may undergo transformations or phase changes due to the thermal energy applied during the boiling process. For instance, continuous exposure of heat on the substrate at decreasing oxygen partial pressures, such as in the degassed operating chamber, can trigger cuprous oxide (Cu₂O) and may be more likely to form as the primary oxide as follows:



On the other hand, some studies [67, 70, 71] presented that initial formation of CuO_2 in hot de-ionized water, while Aljaafari et al. [76] illustrated the formation and growth of a cubic shape cuprous oxide. Additionally, the presence of water vapor or steam during boiling conditions can contribute to the reduction of copper hydroxide ($\text{Cu}(\text{OH})_2$) components. The heat-induced environment, along with the presence of water vapor, can facilitate the redox reaction, converting $\text{Cu}(\text{OH})_2$ into metallic copper (Cu) and water (H_2O) as the following equation describes:



However, $\text{Cu}(\text{OH})_2$ is not stable and can decompose into CuO and H_2O [77], while CuO_2 can transform into CuO in hot water [70] as follows, respectively:



where this reaction can be explained at high temperatures and sufficient oxygen levels shown as following reactions [78, 79]:



The formation of different copper oxide compounds and the alteration of the crystal structure of the existing oxide layer can result in various copper oxide structures on the exposed surfaces. The surface morphology is significantly influenced by the continuous transformation of the oxide layer over time. The oxidation process of copper in de-ionized water involves multiple stages. Initially, there is a complex chemisorption period with surface reconstruction, where oxygen molecules dissociate into atoms

and diffuse across the copper-water interface. At low temperatures, small oxide islands are formed due to slow oxygen diffusion. As the temperature increases, these islands grow by attaching to existing ones rather than forming new ones. Eventually, the islands coalesce to form a uniform oxide layer covering the copper surface, and further growth occurs through diffusion within the oxide layer. Even under atmospheric pressure, the nucleation and growth of the oxide film occur as islands. The formation of small oxide islands in the initial stage may enhance surface wettability due to the presence of non-homogeneous reentrants. However, as the islands continue to grow and coalesce over time, the oxide layer becomes more uniform, potentially resulting in a smoother surface and increased surface temperature due to energy storage in the base surface. However, it is believed that the decrease in boiling performance reaches a plateau once the temperature stabilizes. Additionally, the formation of cubic grains of varying sizes on top of the oxide layer, resembling a cancellous structure, increases the surface roughness. This formation process could explain the local decrease in heat transfer coefficient (HTC) after an initial period of increase, as observed in Figure 20e-h-k.

3.1.3 Effect of number of subsequent CHF-runs and long-term operation

The boiling crisis can instantaneously lead a system transition from nucleate boiling to film boiling, while the temperature of the heated surface can increase dramatically up to 230 - 310°C. Indeed, this can cause low-temperature annealing of the surface, which may cause significant changes in the surface wetting behavior as well as in the initiation of bubbles. As this factor considered, boiling crisis takes an important role in the rate of maximum heat flux, which is the critical design parameter for especially In-Vessel Retention (IVR) process in nuclear engineering [17, 58]. Although the objective of the scenario does not include CHF runs but contains continuous 24 h long-term operation at 45 W/cm², it is therefore interesting to understand the

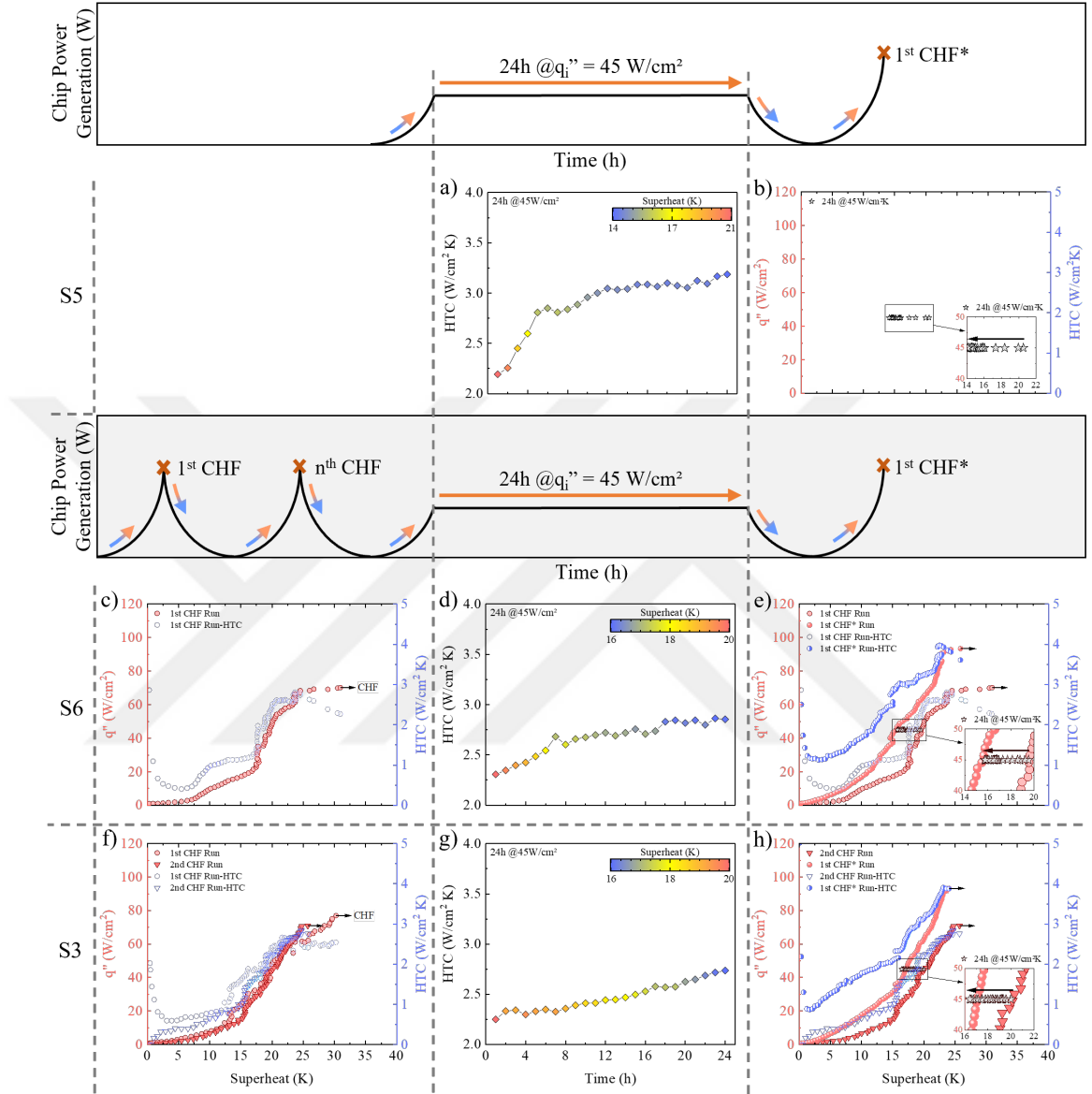


Figure 21: Heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) versus wall superheat obtained for S6 exposed only for 1st CHF run (c), for S3 exposed for 1st and 2nd CHF run (f); temporal change of HTC over 24 h continuous boiling operation conducted at constant heat flux of 45 W/cm^2 for S5 (without any initial CHF run), S6 (after 1st CHF run), and S3 (after 2nd CHF run) with wall superheat indication (a, d, g); shift of heat flux (q'' , red axis) versus wall superheat obtained for S5 (without any initial CHF run) as well as heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) versus wall superheat obtained for S6 (measured after 1st CHF and 1st CHF* run) and S3 (measured after 2nd CHF and 1st CHF* run) including indication (red arrow and stars) of the change of wall superheat within same duration at corresponding constant heat flux level of 45 W/cm^2 (b, e, h).

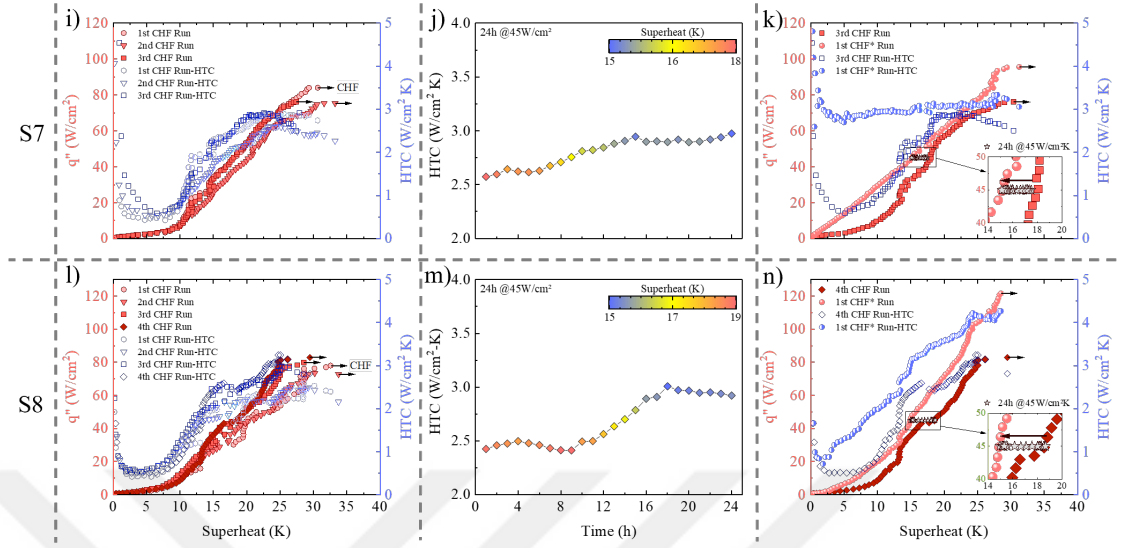


Figure 22: Heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) versus wall superheat obtained after 1st, 2nd, and 3rd CHF runs for S7 (i), and 1st, 2nd, 3rd, and 4th CHF runs for S8 (l); temporal change of HTC over 24 h continuous boiling operation (j, m) conducted at constant heat flux of 45 W/cm²K with wall superheat indication for S7 and S8; comparison of heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) measured after 3rd CHF and 1st CHF* run for S7 and 4th CHF and 1st CHF* run for S8 including indication (red arrow and stars) of the change of wall superheat within same duration at corresponding constant heat flux level (k, n).

individual effect of the continuous long-term boiling operation on heat transfer and surface topography. Moreover, another intention here is also to study the impact of subsequent CHF runs according to the scenario followed by a 24 h long-term operation and final CHF run in order to determine the combined influence of both heating approaches. Figure 21 and Figure 22 comprehensively illustrate the heat transfer performances of the specimens S5 - S8 and show a comparison with S3. The summary of the heat transfer performances under the executed scenarios was presented in Table 4.

The performance of S5 operated in the scenario, which does not include any CHF run, showed a remarkable rise in HTC in the first six hours, while the gradient of enhancement decreased for the rest of the duration. In the end, the maximum HTC resulted in an improvement by 1.0 W/cm²K (45%), and the wall superheat was

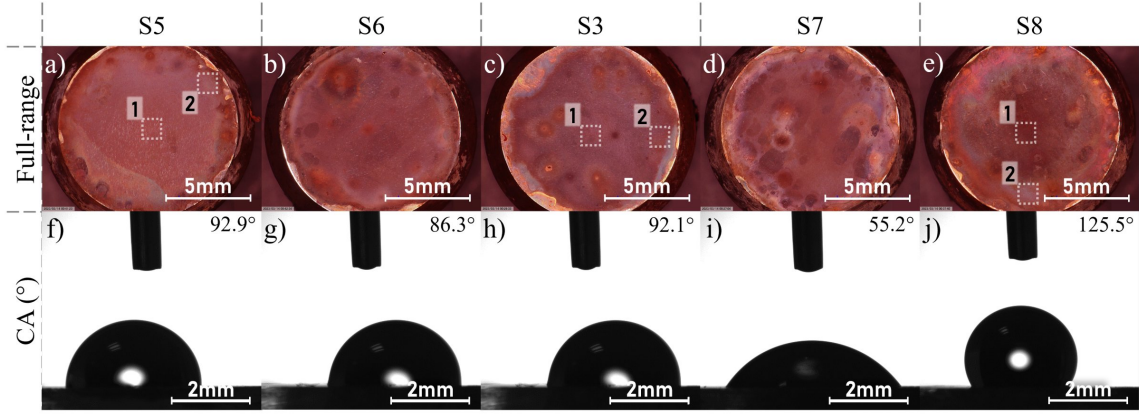


Figure 23: Full range microscopic images with identified areas A-1 and A-2 (a, c, e), and average static contact angle (CA) measurements with the symbolic illustrations (e, f, g, h, j) on the specimens S5, S6, S3, S7, and S8 exposed to boiling, all measurements were done once each series of experiment was accomplished.

reduced by 6.4 K after 24 hours. The sharp decrease in surface temperature could be induced by the possible oxide islands. In the short period, the abovementioned oxide island high possibly cumulated in separate locations by generating presumably biphilic surface conditions, which might enhance the boiling performance [14, 80]. These islands also could provide enormous active nucleation sites by not allowing the thermal boundary layer to form at neighboring locations [81]. However, this surface condition was no more preserved and turned towards a relatively smoother surface condition. The oxide layer grown on the entire surface can exhibit a lower thermal conductivity than the base metal. Indeed, the formation of a thicker oxide layer might cause additional thermal resistance and an increase in wall temperature. In addition, this smooth thermal barrier might have possibly caused local stability on heat transfer performance between the 6th and 8th hours. Once a certain amount of time passed, the decrease in surface temperature continued gradually with the presence of homogenous cubic cuprous oxide Cu_2O grains on S5 (see in Figure 23a-d), which could augment the surface roughness.

The high possibly predicted cuprous oxide microscale grains were observed in similar forms based on SEM images analyzed in the literature [67, 78, 79, 82], whereas

the present XRD pattern (see Figure 24g) of the specimen (S5) could not demonstrate the typical peak positions of $2\theta = 35.9, 36.3, 42.2,$ and 73.2 , which represent the presence of Cu_2O . However, the same surface presented one strong α (alpha) peak at $2\theta = 75.7$ with a β (beta) peak at 75.9 , which might indicate the CuO at $2\theta = 75.5$ (see Figure 24g). At these values, the existence of both alpha and beta peaks represented the emission of both Cu-K alpha and Cu-K beta radiation from the X-ray source. In the experiment, a Cu target with an average wavelength of $1.54 \text{ \AA}$ was used. Therefore, these two peaks do not represent separate Bragg peaks, but rather they both belong to the Bragg peak of the CuO phase. Indeed, the surface was covered by an oxide layer robustly, where the XRD could not detect the pure copper peak positions on calibrated and uncalibrated smooth surfaces. The oxide layer progression was not demonstrated in detail. The disagreement between the images (see Figure 24g-i) and XRD analysis can be nevertheless explained by the transformation of Cu_2O into CuO .

In this scenario, CHF runs were introduced, and heat transfer performances of 2^{nd} CHF runs agreed with 1^{st} CHF runs on S3 and S8, where there is a partial discrepancy for S7 (see Figure 18i). Similar to S1 - S4, 2^{nd} CHF occur earlier with an approximately 6 - 10% smaller value than 1^{st} CHFs for all the specimens. Once comparing the 3^{rd} CHF results, they present a distinctive but better performance than 2^{nd} CHF detected on S7 and S8. In addition, inevitable increases in 1^{st} CHF*s compared to the reference values, which are the final ones of the subsequent CHFs just before the 24 h continuous boiling operations. The data varies between 26 - 33% for S6, S3, and S7.

When evaluating 4^{th} CHF data obtained on S8, this specimen revealed a considerable rising CHF-trend by 6% and 13% greater than 1^{st} CHF and 2^{nd} CHF, respectively. Moreover, 1^{st} CHF* of this surface raised by 47% with an elevated wall temperature compared to the 4^{th} CHF run, which is the reference value (see Figure

Table 4: Summary of CHF and HTC together with performance changes of specimens S5, S6, S3, S7, and S8.

Specimen no. (S#)	Heat flux for 24h operation (W/cm ²)	1 st CHF	2 nd CHF	3 rd CHF	4 th CHF	1 st CHF*	Change for 24 h operation in	
		(W/cm ²)	(W/cm ²)	(W/cm ²)	(W/cm ²)	(W/cm ²)	(%)	(%)
5 $\oplus\oplus$	-	-	-	-	-	-	0.99 45%	superheat (K) -6.40
6	45	●70.1	-	-	-	93.4 33%	0.55 24%	-3.79
3 $\oplus\oplus$	45	○77.0	●70.9	-	-	93.2 33%	0.49 24%	-3.43
7	45	○84.0	75.4 -10%	●76.1 -9%	-	95.6 26%	0.40 16%	-2.36
8 $\oplus\oplus$	45	○78.0	72.8 -7%	79.9 2%	●82.9 6%	121.7 47%	0.50 20%	-3.20

Performance change (%) = 1 - Current CHF / Reference CHF (○, ●)

○ Reference value for the CHF performances

● Reference value for the CHF* performances

* After a continuous 24 h long-term operation at q''_i

$\oplus\oplus$ SEM image and X-Ray diffraction (XRD) analyses are available

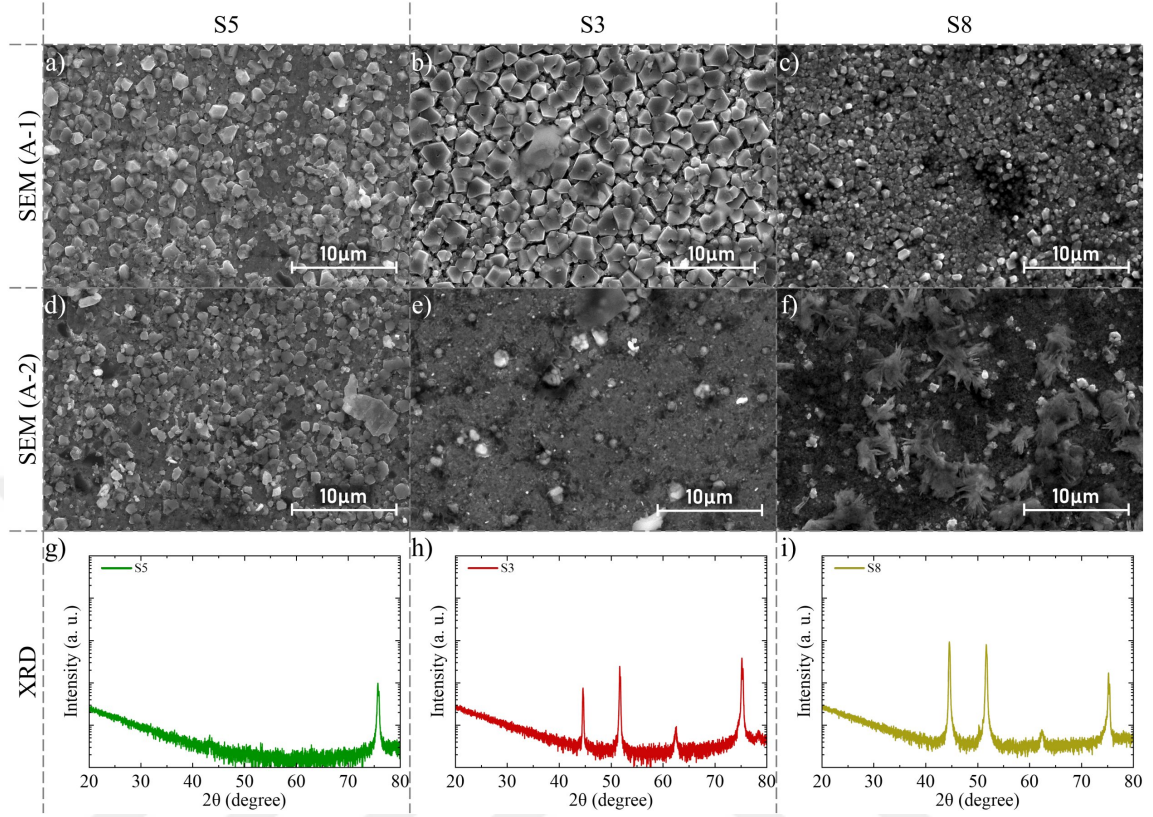


Figure 24: SEM images of the indicated areas shown in Figure 23A-1 (a, b, c) and Figure 23A-2 (d, e, f); X-ray diffraction analyses (g, h, i) on the specimens S5, S3, and S8 exposed to different numbers of subsequent CHF runs, all measurements were done once each series of experiments were accomplished.

22n). This enhancement again could not be associated with the surface wettability, while the oxide compounds were not spread homogenously (see Figure 24c-f), and Cassie's method is favored on S8. In addition, as the surfaces of the specimens were analyzed, a darker region (see Figure 23e) appeared under microscopic images than other substrates, which indicates a weaker harm by the annealing process due to subsequent CHF runs. The SEM images taken at the specimens again supported the existence of a non-porous cuprous oxide layer on the surface characterized like aforementioned by cubic densely packed grains (see Figure 24b). However, the irregularly grown grain clusters formed gullies and simulated micro-cavities. The formation was not supported by the XRD analysis, where the peak position $2\theta = 51.7$ stands for the existence of CuO. The repeating CHF runs provoked leaf-like cupric oxide structures

on A-2 (see Figure 23e), which might be converted from cupric oxides on the S8 with dispersed cubic grains. This similar behavior might also happen on the operated surface of S3, providing the positions of 51.7 and 75.2 degrees that suggest the existence of CuO. Nevertheless, the SEM image on A-1 (see Figure 23b) presented nooky structures in microscales. The boiling curves of the substrates remarkably shifted to the left with increasing HTC after the continuous long-term boiling operations.

In the case of specimen S8, the increase in HTC during the 24 h long-term operation decreased from 45% to 16% as the number of CHF runs rose. This increment by 20% on S8 (see Figure 22m) is larger than the lowest variance of 16% achieved on S7 (see Figure 22j). As the performance of S8 in 24 h long-term operation was considered, it can be deduced that there was a clear local temperature rise after a couple of hours, and this continued after a sharp decrease in surface temperature. The surface temperatures of the remaining specimens (S6, S3, and S7) declined linearly (see Figure 22d-g-j), and even the local surface temperature peaks were not overlooked. The maximum $HTCs$ of the specimens' reference performances (2nd CHF runs) vary between 2.81 and 2.92 W/cm²K for S6, S3, and S7. This value reached as much as 3.25 W/cm²K on S8, whereas the 3rd CHF on the same surface gave 2.98 W/cm²K.

3.1.4 Extended alternating long-term operation and its effect of boiling performance

The stability of CHF and heat transfer coefficient plays a vital role in the maintenance of electronics cooling and nuclear plants. Preventing possible hot spots, drastic jumps in surface temperatures, and material destruction is essential for reliability and lifetime. In design limitations and cost overruns, HTC is also a significant measure and may cost large amount of expenses with inefficient working limitations in the used system, even if it is underestimated incorrectly. In that case, being aware of BHT performance and stability after a 24 h continuous operation is significant for the

maintenance of thermal systems, which may cause a sudden rise in power generation after regular operations. Heat transfer performances of experiments based on scenarios presented in Table 2, as well as an extended long-term operation, are presented in Figure 25 while their results are summarized in Table 5. One scenario was conducted on S9 to test the consistency of subsequent CHF* runs on a surface exposed to a 24 h long-term operation at 45 W/cm^2 . Another one was carried out on S10 to test the consistency of subsequent CHF* runs on a surface exposed to a 24 h long-term operation at 45 W/cm^2 in addition to two successive CHF runs. The first scenario was initiated with continuous nucleate boiling heat transfer at the predefined heat flux on S9. A superior enhancement in HTC with $1.12 \text{ W/cm}^2\text{K}$ and a rise of 49% corresponding to the initial value resulted for that specimen, while a fairly linear surface temperature drop in total by 6.45 K (see Figure 25a) was recorded. The temperature degradation and the rise in HTC showed a nearly identical performance variation with that of S5, which was tested under the same conditions. The subsequent boiling curves after the long-term operations showed an exceptional agreement in each other, while the 2nd CHF* performance is 1% larger than 1st CHF* within the heat flux uncertainty of 3.23 W/cm^2 (see Figure 25b). The maximum $HTCs$ of these runs vary between an extremely narrow range of $4.70 - 4.73 \text{ W/cm}^2\text{K}$. The secondary one was applied solely to S10. The secondary CHF performance exhibits a decrease similar to the rest of the specimens discussed before. Not similar to subsequent performance conformity, 2nd CHF* showed a better performance than 1st CHF* by 5%, while 1st CHF* is 45% better rather than the reference one, 2nd CHF.

Besides, the boiling curves maintained by dynamic heat loads overlaps up to maximum heat fluxes. The HTC enhancement in long-term operation by 24% is nearly identical with the change shown by S3, which was measured at similar operating parameters. The maximum $HTCs$ of these subsequent CHF runs vary between a small range of $3.71 - 3.72 \text{ W/cm}^2\text{K}$. However, this consistency could not be preserved more

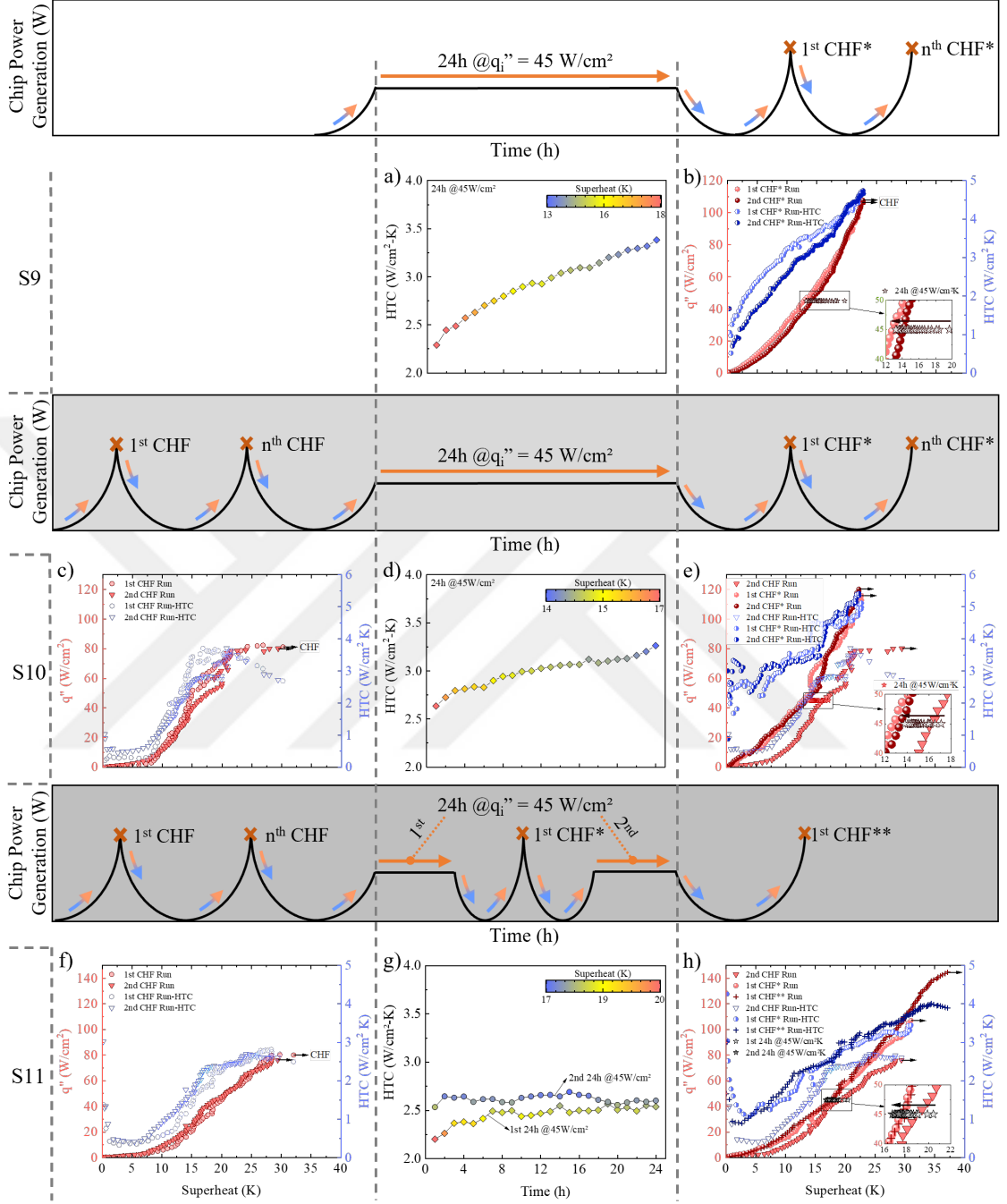


Figure 25: Heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) versus wall superheat obtained for S10 and S11 exposed to 1st and 2nd CHF run (c, f); temporal change of HTC over 24 h boiling operation at constant heat flux of 45 W/cm^2 for S9, S10 with wall superheat indication (a, d); temporal change of HTC over two 24 h boiling operation at constant heat flux of 45 W/cm^2 with one CHF* run (1st CHF*) tested in between for S11 with wall superheat indication (g); heat flux shift versus wall superheat obtained for S9 as well as heat fluxes and HTC s versus wall superheat obtained for S10 and S11 including indication (black arrow and grey stars) of the change of wall superheat within same duration at corresponding constant heat flux level of 45 W/cm^2 (b, e, h).

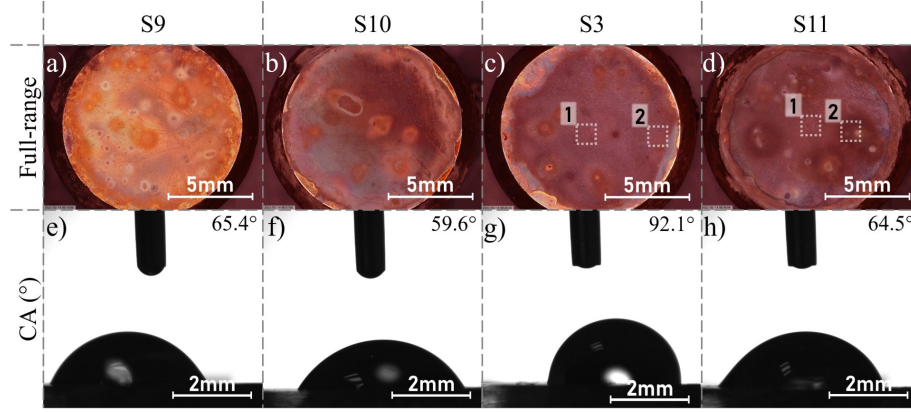


Figure 26: Full range microscopic images with identified areas A-1 and A-2 (a, b, c, d), and average static contact angle (CA) measurements with the symbolic illustrations (e, f, g, h) on the specimens S9, S10, S3, and S11 exposed to boiling.

after the long-term operation, hence CHF^* improved up to critical levels (5.11 and 5.42 W/cm²K), while the number of runs increased.

In order to test the stability of surface performance at dynamic heat fluxes with several continuous boiling sequences, the final scenario was utilized on S11 by heating the surface up to CHF values just after each 24 h long-term operation. The operated surface was devastated more, since prolonged exposure was adjusted. This remarkable destruction can be clearly observed on the entire surface (see Figure 26d). In continuous boiling operations at 45 W/cm², the HTC of the specimen during the first one showed a gradual enhancement by 15%, whereas it is almost five times the change in 2nd 24 h long-term operation (see Figure 25g). CHF^* run and CHF^{**} run showed excellent overlapping at increasing wall superheats up to CHF^* . Moreover, after the long-term exposures, the boiling curves showed linearity rather than the CHF runs on the heat fluxes corresponding to the surface temperatures, as shown in Figure 25h.

CHF^* showed a familiar enhancement by 42% compared to 2nd CHF , while CHF^{**} showed a significant enhancement of heat transfer reaching up to nearly double of 2nd CHF and 35% greater than CHF^* with a prime heat flux of 144.7 W/cm². Similar to

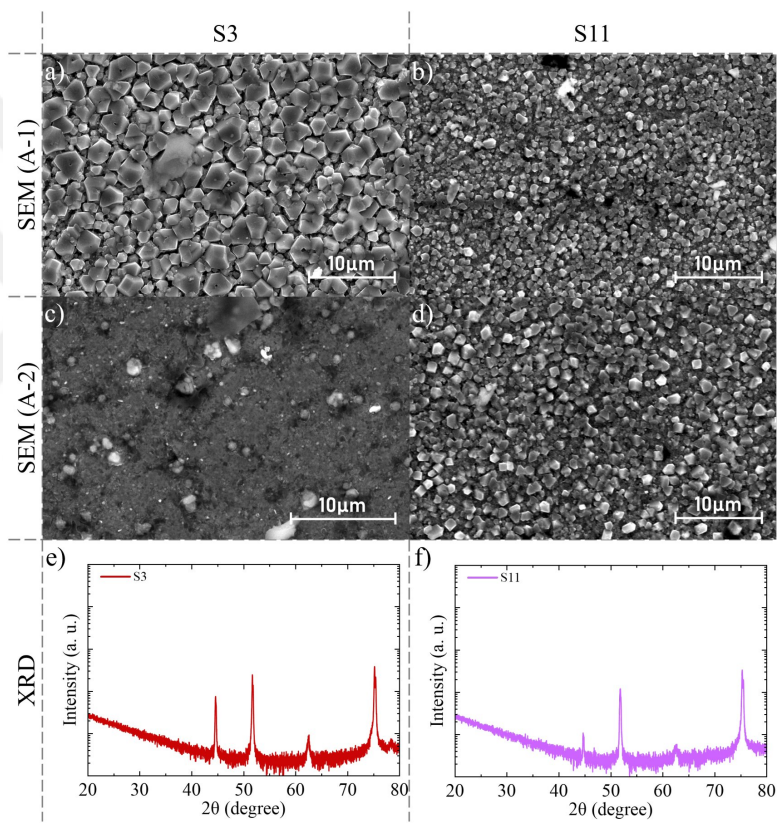


Figure 27: SEM images of the indicated areas A-1 (a, b) and A-2 (c, d) in Figure 26; X-ray diffraction analyses (e, f) on the specimens S3, and S11.

Table 5: Summary of CHF and HTC together with performance changes of specimens S9, S10, S3, and S11.

Specimen no. (S#)	Heat flux for 24h operation (W/cm ²)	1 st CHF (W/cm ²)	2 nd CHF (W/cm ²)	1 st CHF* (W/cm ²)	2 nd CHF* (W/cm ²)	1 st CHF** (W/cm ²)	Change for 1 st /2 nd 24 h oper. in	
							HTC (W/cm ² K)	superheat (K)
9	45	-	-	106.5	107.8	-	1.12 49%	-6.45
10	45	○81.3	●80.1 -1%	111.9 45%	120.3 50%	-	0.63 24%	-3.29
3⊕⊕	45	○77.0	●70.9 -8%	93.2 32%	-	-	0.49 22%	-3.43
11⊕⊕	45	○80.1	●75.4 -6%	□107.0 42%	-	144.7 35%	0.34 / 0.07 15% / 3%	-2.70 / -0.46

Performance change (%) = 1 - Current CHF/Reference CHF (○, ●)

○ Reference value for the CHF performances

● Reference value for the CHF* performances

□ Reference value for the CHF** performances

* After a continuous 24 h long-term operation at q_i''

** After a second continuous 24 h long-term operation at q_i''

⊕⊕ SEM image and X-Ray diffraction (XRD) analyses are available

S10, the largest *HTCs* of the subsequent CHF runs have a rather good agreement. A gradual enhancement by 27% and 16% was observed after each long-term boiling process compared to previous performances. In the final, the maximum *HTC* increased from 2.7 to 4 W/cm²K. This enhancement could adhere to densely packed microcrystals, which might have a great ability to promote bubble growth with the potential to keep trapped vapor in the nano troughs. The homogenously spread nano cubic grains (see Figure 27b-d) are small as the grain size on S3 and S8, exposed to high heat fluxes of 60 W/cm² and most subsequent (4 times) boiling crises, respectively. This can be the explanation for vapor blankets pinning on nucleation sites and spreading on the entire surface, which might be stimulated by the cubic shape grains rather than cancellous and leaf-like formations similar to those presented in Figure 27c.

In addition, it was observed that the cubic shaped crystals were prone to presence and shrink (see Figure 27b-d). Also, the exposure time and surface temperature increase. The XRD resulted in same peak positions at $2\theta = 51.8$ and 75.3 , evidencing the presence of CuO (see Figure 27f) on the fully roofed cubic shaped particles, presumably pointing to those cuprous oxide particles found on S11. Furthermore, in the 1st continuous boiling period (24 h), a type of local decrease was observed in heat transfer performances of the substrates, which supports the similar formation theory occurring on the abovementioned specimens.

3.1.5 Effect of surface roughness

Surface roughness is a key parameter inducing nucleate boiling heat transfer with additional cavities on the heating surface, while nucleation of vapor bubbles occurs randomly due to thermal fluctuations on a smooth surface. As extensively covered in the literature [33, 60, 83, 84], an increase in surface roughness high possibly augments nucleation sites, surface wettability, wicking ability, and surface area.

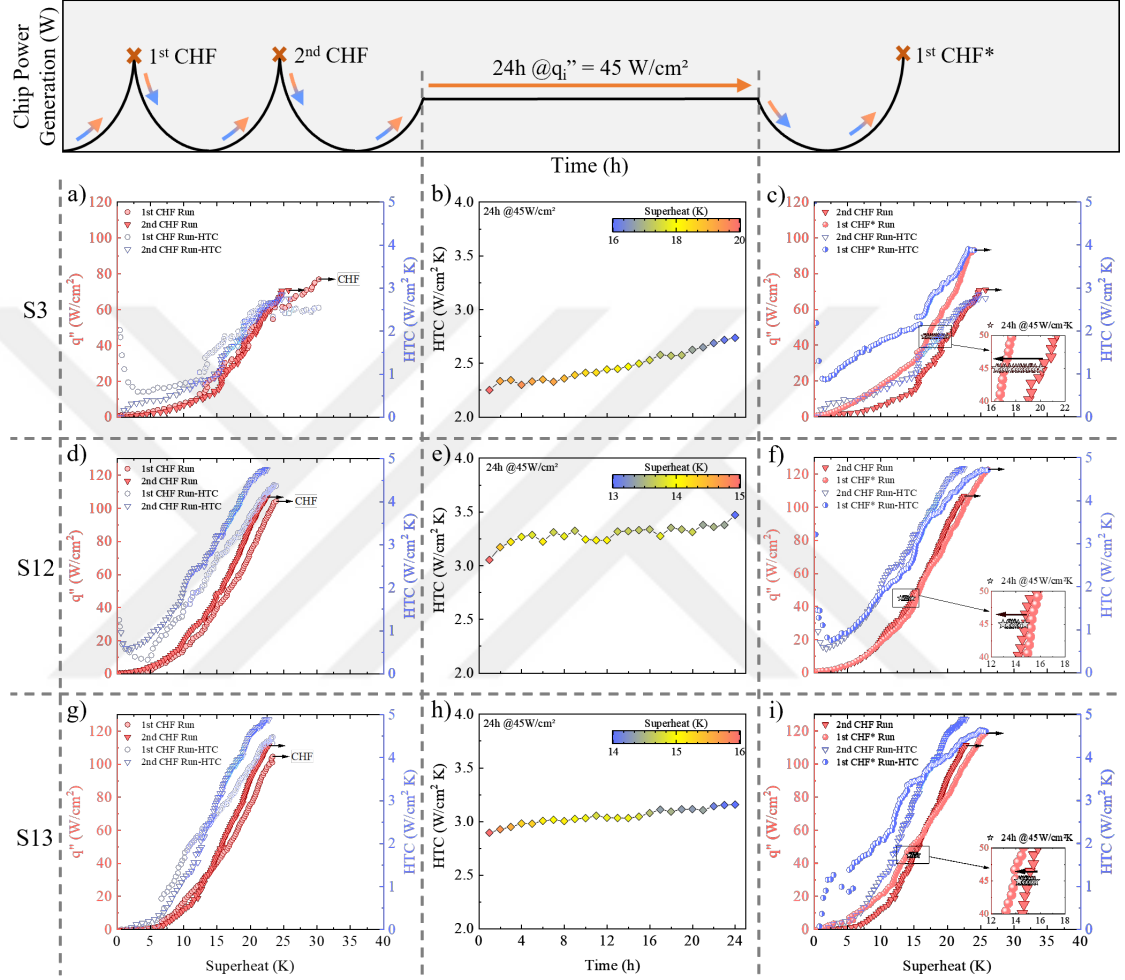


Figure 28: Heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) versus wall superheat obtained after 1st and 2nd CHF runs for S3, S12, and S13 (a, d, g); temporal change of HTC over 24 h continuous boiling operation (b, e, h) conducted at constant heat flux of 45 W with wall superheat indication for S3, S12, and S13; comparison of heat fluxes (q'' , red axis) and heat transfer coefficients (HTC , blue axis) measured after 2nd CHF and 1st CHF* run for S3, S12, and S13 including indication (red arrow and stars) of the change of wall superheat within same duration at corresponding constant heat flux level (c, f, i), the surfaces of specimen S3 was mirror-like polished, while those of specimens S12 and S13 were treated with silicon carbide sand papers with #320 and #120 grit sizes, respectively.

In order to assess the influence, whether the oxide layer or selectively generated surface roughness are dominating the heat transfer enhancement in boiling, or to what extent their combination is determining the overall process, three surfaces with several surface roughness i.e., S3, S12, and S13 that have been exposed to the illustrated scenario. Specimen S3 had a mirror-like polished surface condition. The surface of specimens S12 and S13 were manufactured with similar methods applied for S3; however, after their surfaces were intentionally manipulated by using silicon carbide abrasive papers with a grit size of #320 and #120. The boiling performances are presented in Figure 28 showing the results of two successive CHF runs, a 24 h long-term operation at 45 W/cm^2 , and a final CHF run. Other than S3, the CHF curves of S12 and S13 started to differ, where the 2nd CHF values slightly increased and shifted to the left compared to the initial ones (see Figure 28d-g).

1st CHF of the coarse surfaces (S12 and S13) showed nearly 35% better performance compared to polished one with about 104.5 W/cm^2 , while the maximum *HTC* of S12 is nearly double the *HTC* of S3. In contrary to the plain surface, the 2nd CHF of S12 increased marginally by 7%, whereas a noticeably smaller value resulted on S13 with 2%, which was embossed by deeper scratches. Furthermore, it seems that providing the heater surface with initial micro-roughness yields, if we take the liberty, a rather linear propagation for CHF and *HTC*, respectively. The 24 h long-term operation tests revealed that the *HTCs* of S12 and S13 increased in any case within a tight range of 9 - 13% (see Figure 28e-h), but not as much as the polished surface (22%). In final, 1st CHF* values increased up to a certain heat flux plateau of 120 - 123 W/cm^2 with 7% (S12) and 15% (S13) enhancements compared to reference performances (2nd CHF). On the opposite to the mirror-like polished surface, prone to create a remarkable change in boiling performance, the maximum *HTC* of S12 and S13 preserved the consistency in a sequence of CHF runs and in CHF* runs compared to reference performance, too.

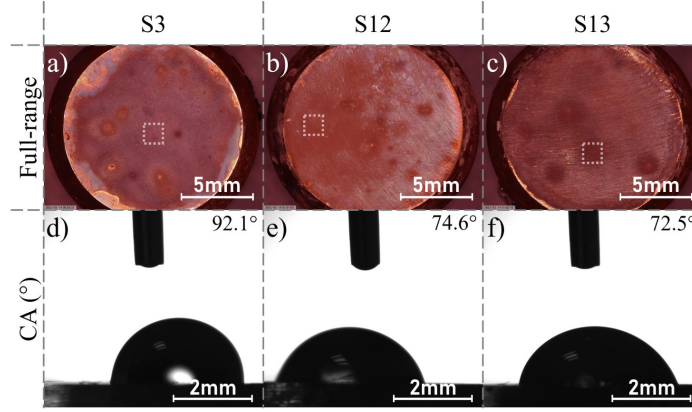


Figure 29: Full range microscopic images with identified areas A-1 and A-2 (a, b, c), and average static contact angle (CA) measurements with symbolic illustrations (d, e, f) on the specimens S3, S12, and S13 exposed to boiling.

Overall, we think that the boiling performance of the exposed rough surfaces was not affected significantly by the oxide layer formations, while a great agreement on CHF values after boiling crises and long-term boiling operations exists. Measurements on static contact angles showed smaller values for S12 and S13 compared to S3, while macroscopically, the surface oxidation was more apparent on S3 (see Figure 29a-b-c). Cubic shaped oxide compounds raised homogenously in the scratches like seeded along the micro-channels as symbolically presented in Figure 30e-f. S12 was irritated by a #320 grit paper and smoother than the S13 treated by a #120 grit paper. Hence, the micron-sized grains were densely packed, while the micro-particles covered the entire surface by filling the microchannels on the untreated surface (see Figure 30e) on S12. Different from the other surfaces, on this surface, an improbable peak position was observed at $2\theta = 44.5$ degrees (see Figure 30h), where it does not match with the International Centre for Diffraction Data (ICDD). This could be reasoned by the possible crystal lattice defects, size, and shape effects in micro scales, where the material is pure and measured in high resolution. The propagation on the rough surface might bring all the indentations to the same level and presumably decreased the surface roughness. The micro-channels filled by the

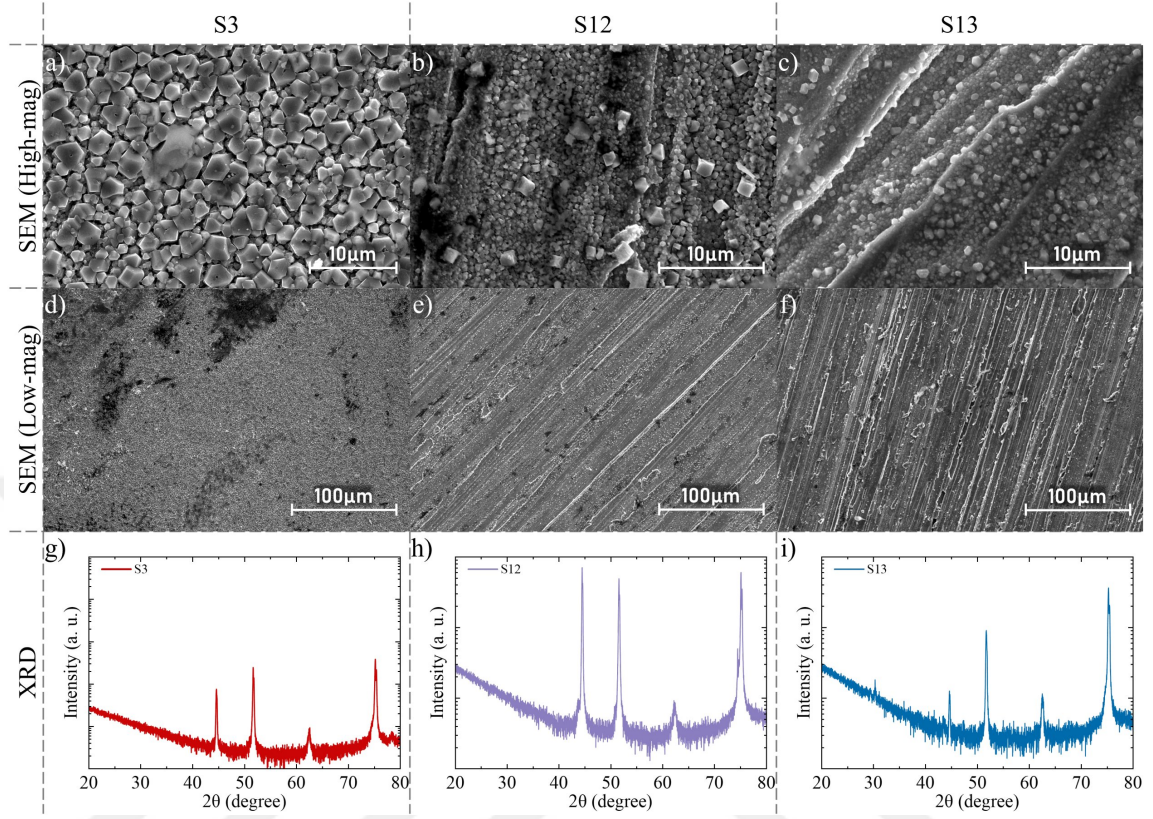


Figure 30: SEM images of the indicated areas A-1 (a, b, c) and A-2 (d, e, f) in Figure 29; X-ray diffraction analyses (g, h, i) on the specimens S3, 12, and S13 with different roughnesses.

micro-grains could enhance the friction effect and decrease the pressure difference by bubble departures, presumably costing a gradual decrease in maximum heat flux and heat transfer coefficient (see Figure 30b). On the other hand, similar grains rose in the deeper micro-channels on S13 (see Figure 30c). Different from S12, the presence of micro-particles did not defect the surface roughness and most probably provoked the bubble growth with additional vapor trapping effect in re-entrants. Thus, HTC and CHF performance of the exposed surface advanced at mid-range heat fluxes, where this effect reversed after a certain level. CHF and HTC values and their relative changes are listed in Table 6.

Table 6: Summary of CHF and HTC together with performance changes of specimens S3, S12, and S13.

Specimen no. (S#)	Heat flux for 24h operation (W/cm ²)	1 st CHF (W/cm ²)	2 nd CHF (W/cm ²)	1 st CHF* (W/cm ²)	Change for 24 h operation in HTC (W/cm ² K) (%)	superheat (K)
3 $\oplus\oplus$	45	o77.0	●70.9 -8%	93.2 32%	0.49 22%	-3.43
12 $\oplus\oplus$	45	o104.7	●112.2 7%	119.9 7%	0.26 9%	-1.12
13 $\oplus\oplus$	45	o104.3	●106.8 2%	122.9 15%	0.42 13%	-1.78

Performance change (%) = 1 - Current CHF/Reference CHF (o, ●)

o Reference value for the CHF performances

● Reference value for the CHF* performances

* After a continuous 24 h long-term operation at q''_i

$\oplus\oplus$ SEM image and X-Ray diffraction (XRD) analyses are available

3.2 Boiling Heat Transfer Enhancement on Micro-drilled Surfaces

The specimens used as test surfaces were abraded by up to #7000 grit water waterproof and dry sanding sheet. The heat transfer surfaces were polished to achieve a defined surface finish. Subsequently, a micro-drill process was applied on two sample surfaces to obtain artificial cavities on pre-defined locations as depicted in Figure 31. With a drill-to-drill space of 1 mm, the cavities were distributed in a hexagonal pattern yielding a drill density of 75 drills/cm². Two sizes of micro-tapped blind holes were adopted, 200 μm and 700 μm , whereas the depth of the drills was kept at about 300 μm and 400 μm , respectively. The geometric sizes and the resulting liquid-solid contact angle (CA) were measured as shown in Figure 31. In addition, the contact angle of measured at the bare surface was $88.3 \pm 1^\circ$.

The experiments have been conducted on the plain surfaces, MD02-1, and MD07-1 under wide range of pressure and subcooling conditions as depicted in Table 7. The

Table 7: Experimental micro-drilled surfaces and operating conditions.

Specimen no. (S#)	Drill to Drill Distance (μm)	Drill Diameter (μm)	System Pressure (bar)	Liquid Subcooling Temperature		
				0 K	5 K	10 K
Smooth	-	-	1.00	+	+	+
			1.25	+	+	+
			1.50	+	+	+
MD02-1	1000	200	1.00	+	+	+
			1.25	+	+	+
			1.50	+	+	+
MD07-1	1000	700	1.00	+	+	+
			1.25	+	+	+
			1.50	+	+	+

+ Performed experiment

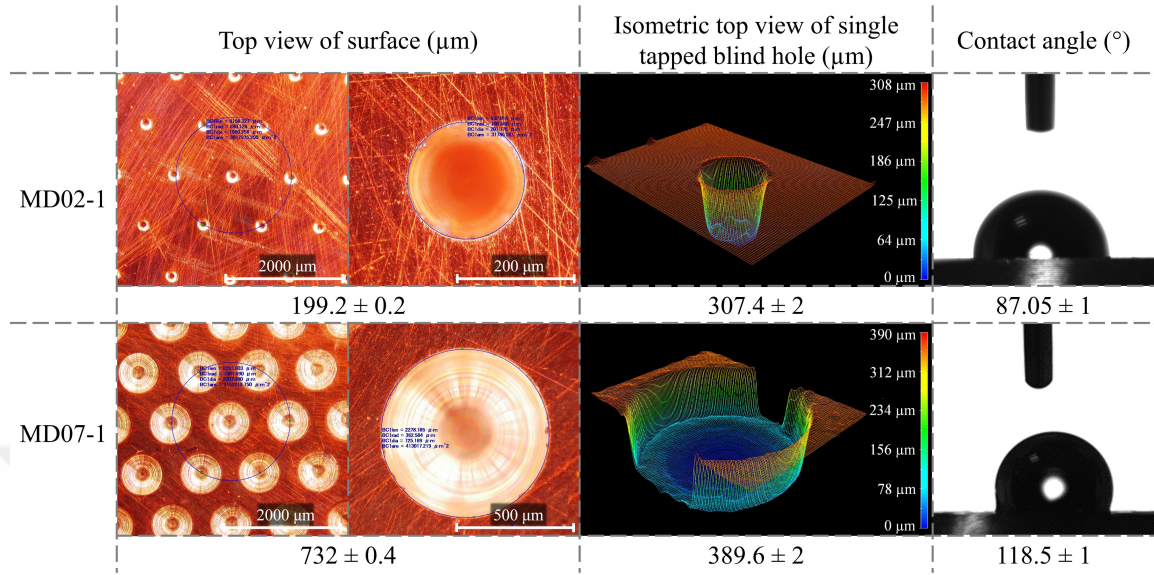


Figure 31: Microscopic surface characterization and contact angle (CA) images of micro-drilled surfaces.

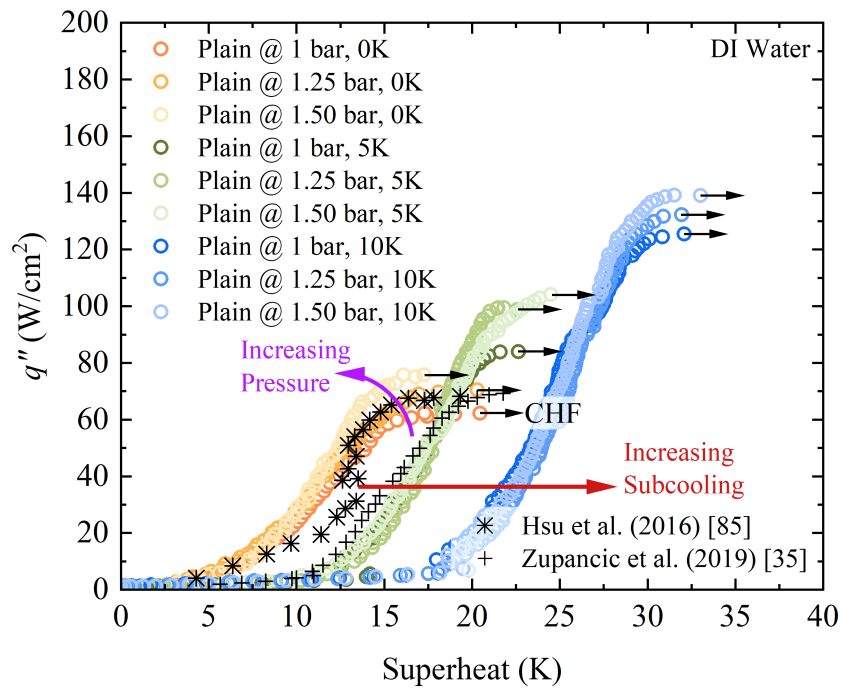


Figure 32: Boiling curves at 0, 5, and 10 K subcooling temperatures, and 1.0, 1.25, and 1.50 bar operating pressures on bare surface compared with literature and correlations.

initial tests were conducted on the plain surface in DI water at saturated temperature and atmospheric pressure. The CHF of the bare surface (62.2 W/cm^2) has been compared with the heat transfer performance of bare surface with the experimental results by Hsu et al. [85] and Monze et al. [35], a good consistency on heat flux values concerning the superheat increase was obtained under similar operating conditions as shown in Figure 32.

Zhang et al. [86] presented that the boiling curves have shifted to the right as the subcooling temperature increases from 0 to 20 K. Pattanayak and Kothadia [87] have stated that as the subcooling increases, the magnitude of CHF also increases for subcooling and heat rate during the nucleate boiling. The exemplary results in the form of boiling curves demonstrate a trend of shifting to the right within a small amount of surface temperature increase. The boiling heat transfer of the plain surface has doubled as the subcooling temperature increase. Also, the rest of the surfaces

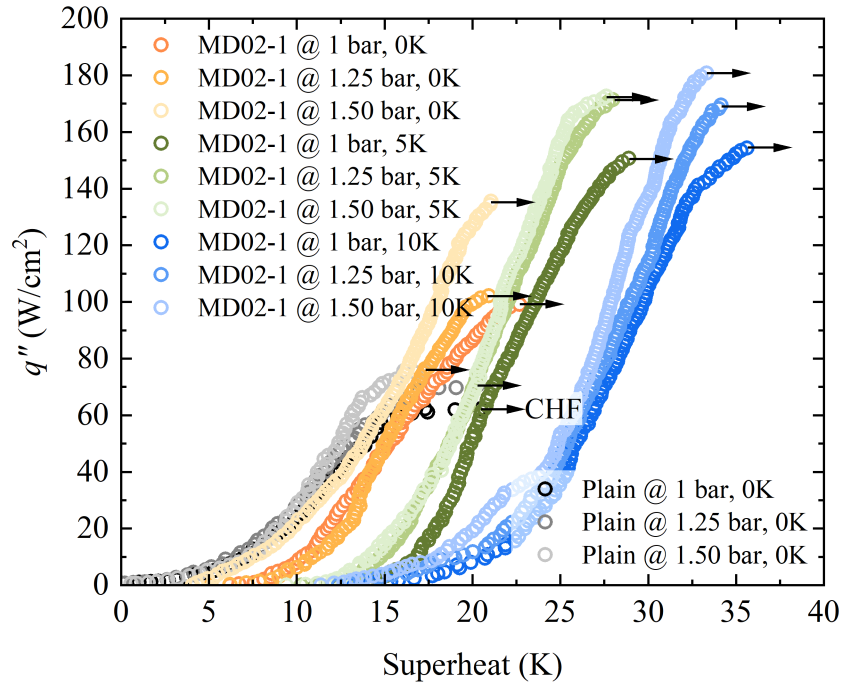


Figure 33: Boiling curves at 0, 5, and 10 K subcooling temperatures, and 1.0, 1.25, and 1.50 bar operating pressures on MD02-1 compared with bare surface.

showed a considerable enhancement of 56% and 24% on CHF values compared to saturation performances of MD02-1 (see Figure 33) and MD07-1 (see Figure 34), respectively. The reason for this relative increase can be strongly attributed to a larger number of nucleation sites as well as to the enlarged liquid/vapor heat transfer interface area at the so-called 3-phase contact line [88]. Another reason might be the enhanced capillary suction of bulk liquid towards the surface. The minor decrease in the heat transfer enhancement of surface MD07-1 for subcooling temperature may be due to larger bubble sizes at the cavities leading to early CHF. Therefore, subcooling has fewer effects on the surface MD07-1 compared to the rest of the surfaces. In another standpoint, this might be due to the large-sized cavities, which may lead to the entrapment of larger amount of vapor inside the drills, hence to an early onset of CHF. Indeed, MD02-1 has an uncontradictable advantage over MD07-1 with the smaller size of artificial cavities at subcooling temperatures with the exception of

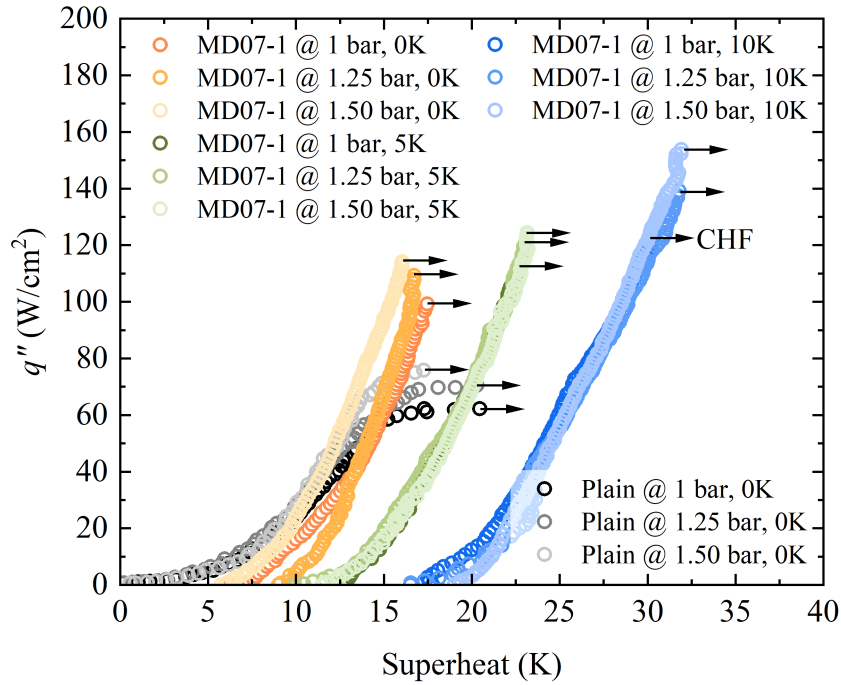


Figure 34: Boiling curves at 0, 5, and 10 K subcooling temperatures, and 1.0, 1.25, and 1.50 bar operating pressures on MD07-1 compared with bare surface.

saturated conditions as shown in arrows in Figure 35. The contribution of pressure on nucleate pool boiling heat transfer results as CHF increase at a few more or less superheat. In case of MD02-1 at 1.5 Bar, CHF showed an amount of growth over 37% at saturation temperature, while the increase in CHF for MD07-1 at 1.5 bar was 25% at 10 K subcooling temperature compared to atmospheric condition. Similarly, for a larger subcooling, an increase in CHF for both surfaces could be obtained, however, the rate of improvement decreased. Corresponding to the results, the overloaded pressure affects the HTC performance negatively, whereas it shifts the boiling curve to the left at the high heat fluxes ($>60 \text{ W/cm}^2$). Dahariya and Betz [89] also noticed a decreasing degree of wall superheat while presenting the enhancement of NBHT with increasing pressure. This can be explained by the thermal boundary layer and the merging of vapor trains propagating vapor explosion resulting in CHF enhancement

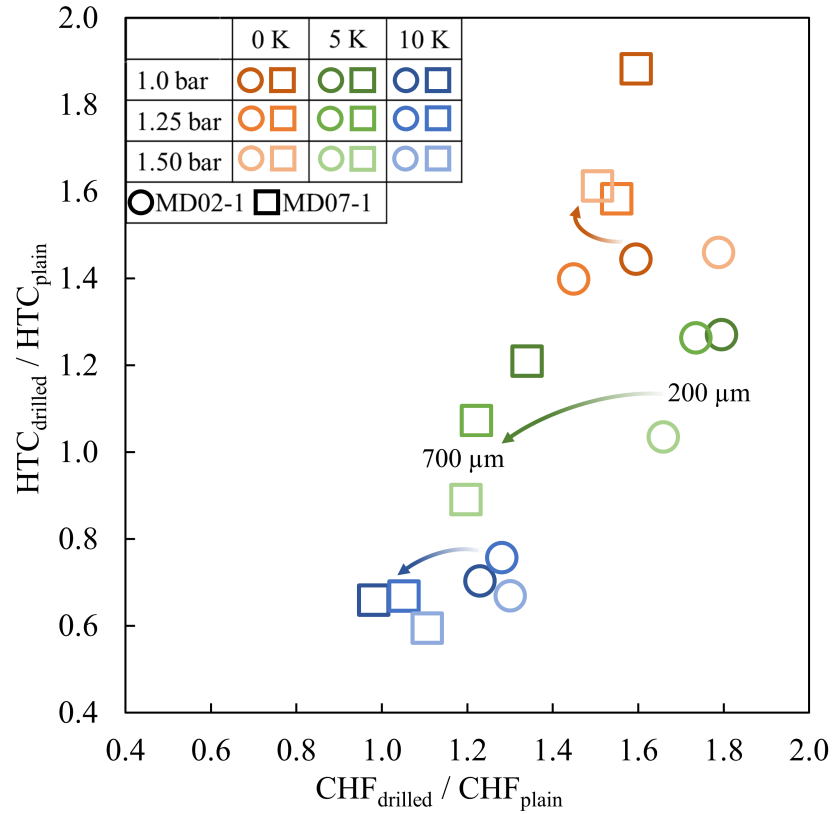


Figure 35: Dimensionless CHF and HTC values of the results referred to the plain surface data.

where the surface tension of the liquid is at different pressures.

The microcavities due to surface polish and artificial cavities by micro-drill machining provide a great opportunity for bubbles so they can depart more frequently with smaller sizes from the heating surface, especially at high subcooling temperatures (see Figure 36). Despite to this advantage on heat transfer, subcooling is shown hardly affect the bubble growth regime, because it has been observed that as the subcooling level increases, bubble nucleation decreases due to the reduced evaporation latent heat, and this also damages HTC of the boiling surface. This effect has been demonstrated by decreasing HTC and CHF ratios of micro-drilled surfaces on bare surface to their half of values (see Figure 35). In other words, the artificial cavities by micro-drilling have no more external impact on heat flux, whereas they can be a threat for HTC as subcooling temperature increases. In addition, as obviously shown in the photographs taken in the subcooled liquid (see Figure 36), the diameters of the micro-drills control the bubble departure sizes and frequency where from MD07-1 to MD02-1, the bubble sizes are smaller, and frequencies are higher.

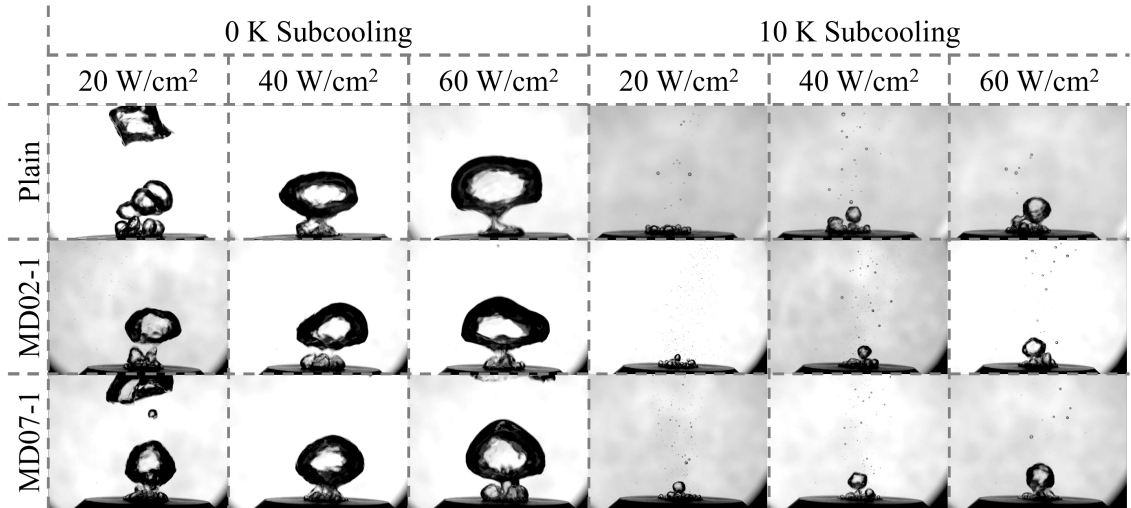


Figure 36: Departure of growing bubbles from surfaces of plain, MD02-1, and MD07-1 samples at 0 K and 10 K subcooling, and heat fluxes of 20 W/cm², 40 W/cm², and 60 W/cm².

3.3 Boiling Heat Transfer Enhancement on Additive Manufactured Micro-channels

The test samples were manufactured with an outer diameter of 10 mm and 25 mm in height. 6082 aluminum alloy (AlSi1MgMn) and AlSi10Mg fabricated via AM using powdered feedstock were preferred for a uniform temperature distribution over the cast and AM surfaces, respectively. Two distinctive methods were used in the manufacturing process: computer numerical control (CNC) machining and additive manufacturing. One of the six specimens was manufactured in a lathe machine and fractionally abraded by sandpapers and water-based monocrystalline diamond suspension to get a well-defined mirror-like finished surface by reducing the size of scratches and reentrants presenting $1.23 \mu\text{m}$ average surface roughness (R_a). This surface finishing was repeated for one AM plain surface (Figure 37a) and achieved $R_a = 0.07 \mu\text{m}$. The remaining surfaces consist of one plain (Figure 37b) and three different microchannel additive manufactured surfaces (Figure 38a-b-c).

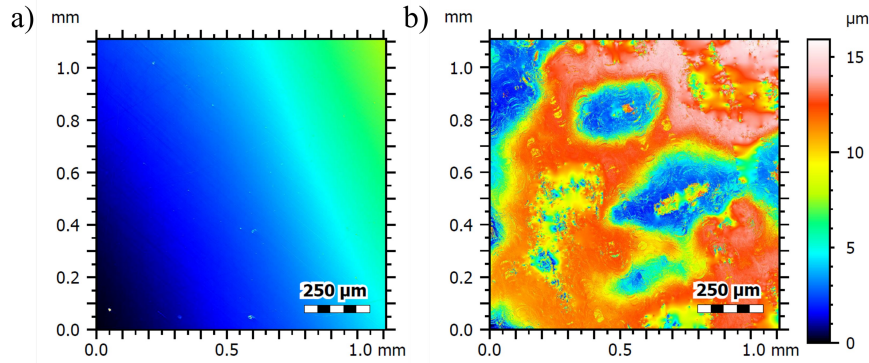


Figure 37: Confocal analysis: a) Surface AM0P, after polish ($R_a = 0.07 \mu\text{m}$); b) Surface AM0, before polish ($R_a = 1.23 \mu\text{m}$).

The microchannels were manufactured as mono-blocks with the specimens, and their geometries are rectangular (MC1), V-shape (MC2), and inverse V-shape (MC3), as shown in Figure 38. The geometric dimensions of MC1 were set to be $400 \mu\text{m}$ for height and width. For the microchannel surface of MC2, the V-shape opening is

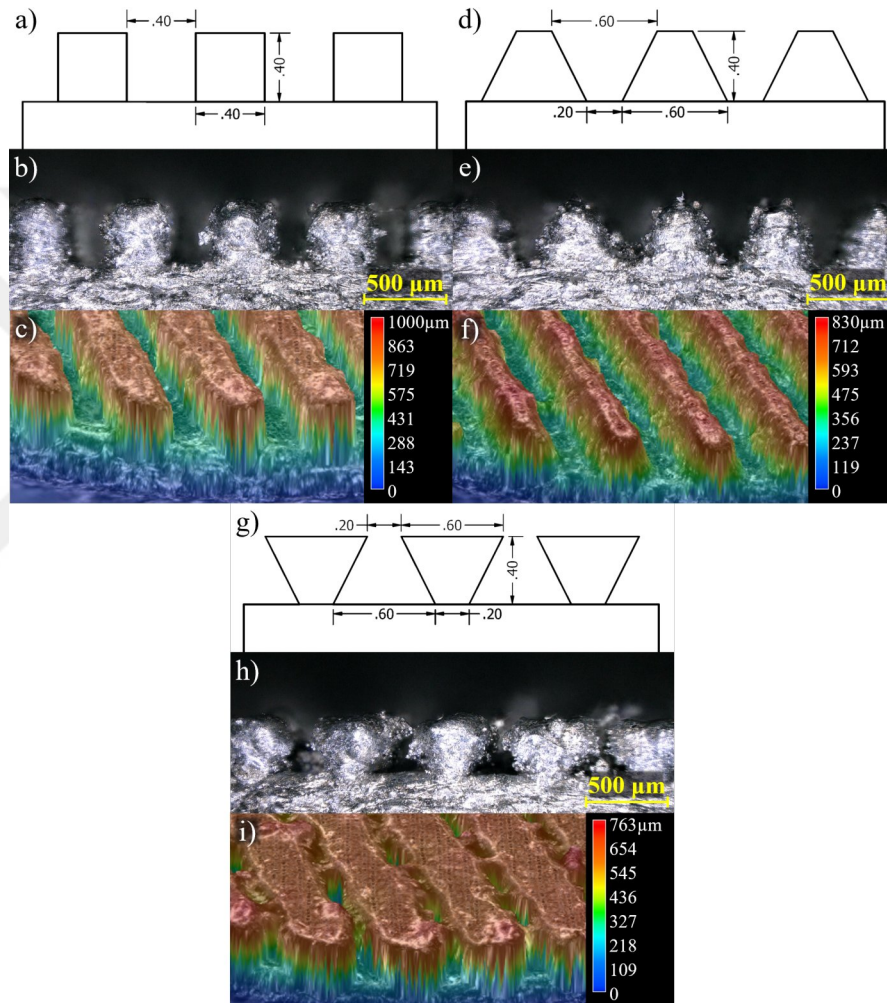


Figure 38: Geometrical dimensions, section view, and confocal analyses of the additive manufactured surfaces: a-b-c) MC1, d-e-f) MC2, and g-h-i) MC3 microchannel surfaces. The scale bars on section views refer to 500 μm .

600 μm , while the base was designed to be 200 μm . MC3 was configured to have the exact sizes as MC2, though inversely oriented. Once the particle size distribution and potential protrusions in the production were considered, the channel sizes were limited in order to preserve the predefined channel geometries and prevent the closure of the channel mouths, which can obstruct the vapor escape. In addition, two plain samples (unstructured) were produced, one in a lathe machine (LM) within CNC configuration and another one with AM (AM0). The latter was tested with and without polishing on the surface. The production of the five AM surfaces was employed via a Renishaw AM500Q Flex, which is a laser powder bed fusion (L-PBF) machine. AlSi10Mg powder provided by Carpenter Additive with a particle size distribution of 20 - 63 μm was used. The machine is equipped with a quad laser system, and the laser assignment during fabrication was one laser per part. The key process parameters recommended by Renishaw Inc, which are a laser power of 350 W, laser speed of 1.8 m/s, hatch spacing of 0.1 mm, scan rotation of 67 $^\circ$, and layer thickness of 30 μm with a 5 mm wide stripe scanning strategy were preferred. The production includes an exposure time, i.e.: the time spent by material melting, and a dosing time, i.e.: the time spent by the machine spreading the new powder for the next layer. The exposure time of the microchannel geometries of MC1, MC2, and MC3 over a 10 mm diameter plain surface were approximately 18.6 s, 17.4 s, and 19.7 s, respectively. Once the total exposure time is considered, the total production time of each microchannel geometry on a single surface is about 2 minutes, where the system has four lasers. Specimens were printed on pin support structures and removed carefully with a chisel and hammer to prevent damage to the specimen surface. Surface quality characterization of test specimens was conducted on a Keyence VHX-6000 optical microscope (1/1.8-inch CMOS image sensor Virtual pixels: 1600x1200).

Boiling heat transfer was first measured in dielectric liquid HFE-7100. Two subsequent increases in heat flux were conducted up to the CHF point for the experimental

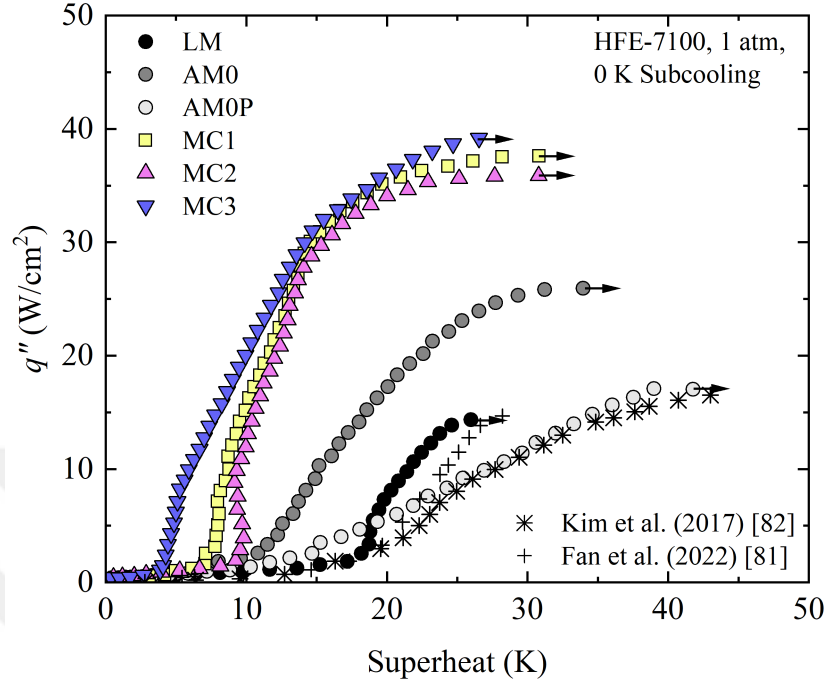


Figure 39: Boiling curves for additive manufactured (AM) surfaces in saturated HFE-7100 at atmospheric pressure.

surfaces and showed identical results for each surface condition. The boiling performance of the first two experiments on LM and AM0P manufactured surfaces showed a variety, although their surface conditions are nearly identical. The LM surface has dissipated 14.4 W/cm^2 , while AM0P performed about 2.7 W/cm^2 more. Once the heat transfer performances of the LM and AM0P were compared with the literature, both were observed to demonstrate a strong agreement with experimental data by Fan et al. [90] and Kim et al. [91], as depicted in Figure 39. The AM0P surface heated up and showed weak *HTC* after a certain heat flux. Next, the AM0 test was executed and showed 52% enhancement in CHF, where its *HTC* doubles (see Figure 40). The noticeable augmentation on CHF can be attributed to additional liquid supply by increased surface roughness and wettability, while this factor delays the dry-out of the liquid sublayer to higher surface temperatures [59]. In fact, it's believed that capillary wicking is more intensive in the micro-geometries; however, prediction of the

surface roughness is a challenging process with contact angle measurement, where the test droplet is a highly wetting and volatile dielectric liquid [92, 93]. In addition to surface wettability, likely the higher nucleation site of the plain surface, which has 17 times larger indentations (see Figure 37), may explain the *HTC* increase with the increasing heat flux. High-speed records also point out increased bubble departure with smaller diameters (see Figure 41), which contributes to liquid replenishment on the densely evaporated plain surface rather than the polished one [94]. As the cavity size increases, the inertial forces decrease, where buoyancy and surface tension play a dominant role on bubble departure, and this explains smaller bubble departure from the rough surface [95]. The micro-pores and scratches were flooded with the highly wetting dielectric liquid, and a wall temperature was required for the initiation of bubbles. In this condition, trapped vapor in the microcavities (see Figure 37) provides an advantage to decrease the surface temperature of boiling incipience [96]. In summary, the rough plain surface shows the same heat flux performance at lower wall superheats compared to the polished one.

In general, microchannel surfaces showed an extraordinary enhancement on BHT, where their trends are in a similar direction. MC1 enhanced CHF by 121% compared to AM polished surface, whereas MC2 showed 110% enhancement. On the other hand, MC3 showed the best performance with 130% and 386% enhancement on CHF and *HTC*, respectively. We assume that the CHF enhancement has been maintained by the channel geometries due to their strong suction-evaporation mechanism. This capillary-scale pumping action worked in the channels where the pressure goes down, introducing a liquid suction through the inactive pores. As the channel space was filled with vapor blanks, this prevented the liquid suction stimulating CHF [97]. However, CHF on the microchannels shows variety even though the channel sizes are identical, which can direct to channel geometry. At high fluxes, vapor bubbles growing from neighbor channels are more prone to coalescence for MC2. This provides larger bubble

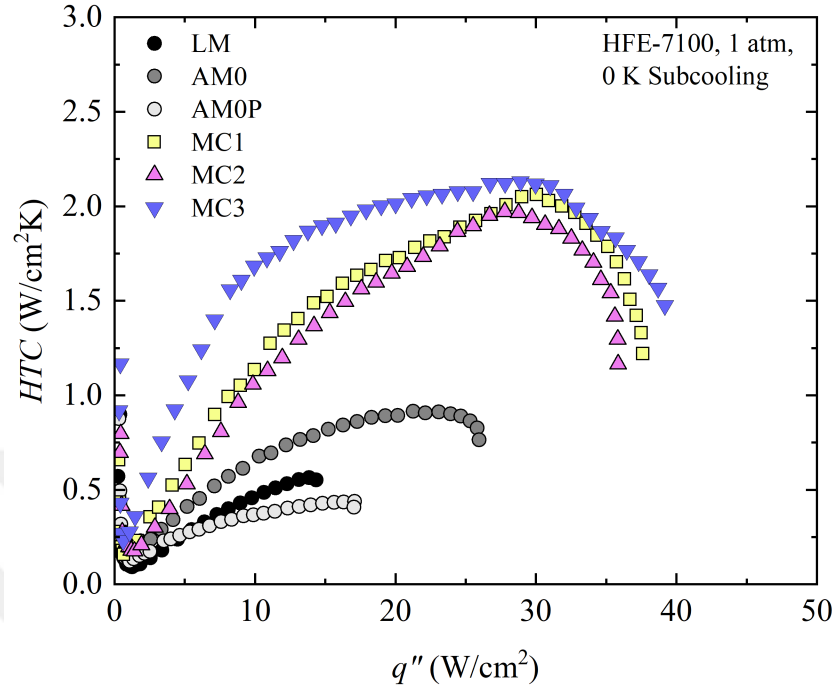


Figure 40: Variation of heat transfer coefficient with heat flux for additive manufactured (AM) surfaces.

departures over the heating surface and leads to early CHF. In addition, larger bubble departures have been observed with lower frequency at low and intermediate heat flux levels (see Figure 41) as in Singh et al. [98]. For a wider channel opening, the bubble tends to acquire the wall profile shape by growing laterally along the channel height.

This might be caused by the high surface tension force that confines the bubble to adhere more strongly to the solid wall. Then, it can reduce the bubble growth rate and frequency of bubble formation where the bubble is held in place by the surface tension force rather than the buoyancy. Indeed, the bubbles may experience more resistance on the wall resulting in a higher surface temperature for boiling incipience and a lower HTC [50]. On the other hand, MC3 enables early incipience of bubbles with small size taking off from the surface with higher frequency at low heat fluxes, which brings an advancement on HTC rather than other channel geometries (see Figure 40). Hubner and Kunstler [44] and Rajulu et al. [47] have also demonstrated

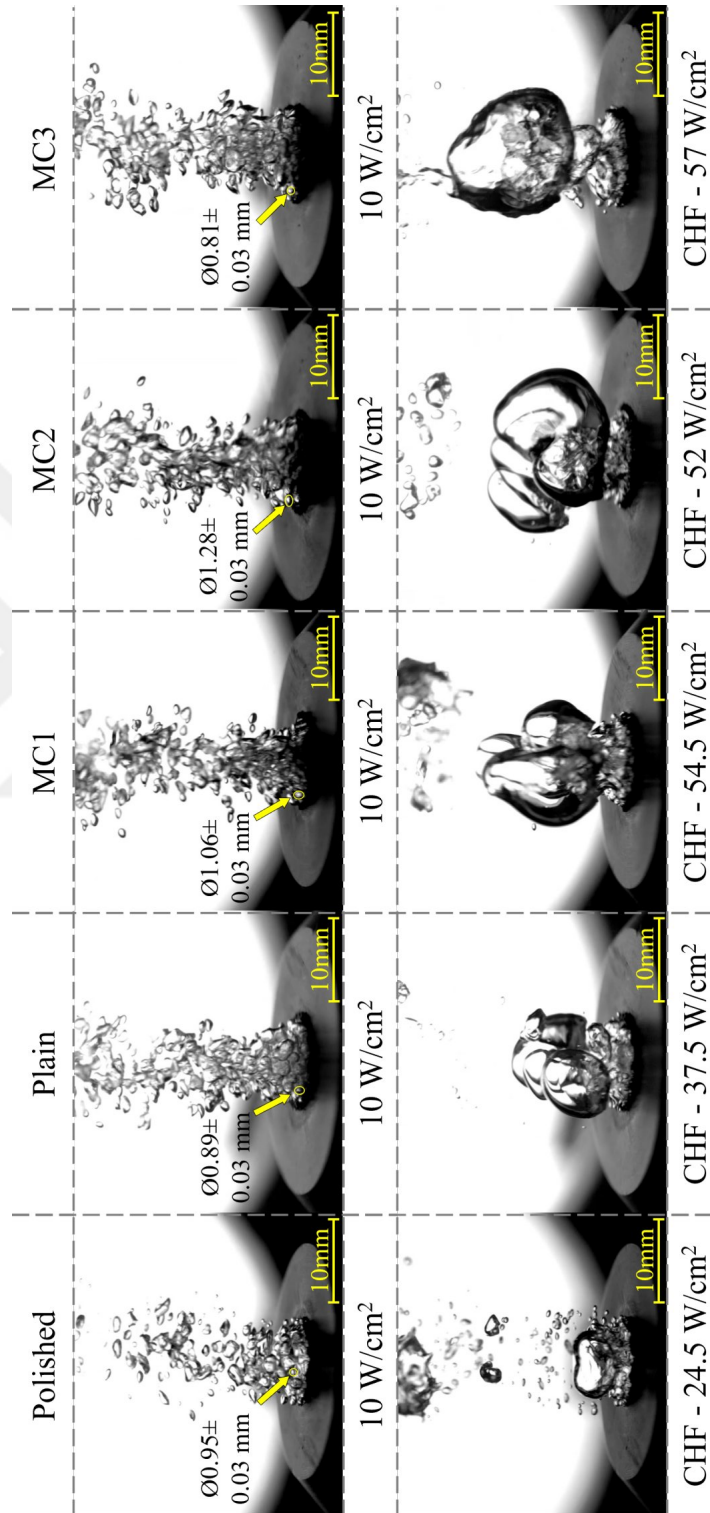


Figure 41: Bubble departures growing from AM0P, AM0, MC1, MC2, and MC3 samples at 10 W/cm², and near CHF.

similar advancement with re-entrant cavity surfaces in a significant manner. The ever-narrowing upward geometries separate the bubble vapor escape routes and liquid supply pathways while preventing bubble merging at high heat fluxes. Thus, hot spots cannot easily develop, and CHF condition does not occur at the heat fluxes as soon as other geometries [99]. The HTC of the microchannel surfaces increased in the discrete bubbles and fully developed nucleate boiling regions, at low and intermediate wall superheats. The number of active nucleation sites increases as heat flux increases in the discrete bubble region, and in the fully developed nucleate region, all potential nucleation sites become active. Beyond this point, a further increase in heat flux provokes lateral coalescence of growing and departing bubbles, which causes inevitable dry-out spots. These continuous large spots create instant but highly frequent thermal resistance, increasing surface temperature and decreasing HTC [100]. These differences in boiling performances of the microchannels demonstrate that additional surface area is not a simple factor where the channel geometry has a considerable effect.

CHAPTER IV

CONCLUSIONS

A detailed experimental study was carried out on copper and aluminum substrates with distinctive boiling scenarios. To detect the potential oxidation effect on boiling heat transfer, several operation scenarios comprised the objectives such as the effect of heat flux rate, successive boiling crisis, long-term operation, and surface roughness. For each target, experimental results have been evaluated separately. Moreover, the stability of the heat transfer performances was investigated, while smooth and rough surfaces were tested in saturated de-ionized (DI) water at atmospheric pressures. In addition, an experimental study has been performed with DI water to understand two fundamental characteristics which are subcooling and pressure on a copper plain and micro-drilled surfaces. In final, an experimental study presented the capability of additive manufacturing on surface structuring in micro-scales and its contribution to boiling heat transfer. Experiments were performed in HFE-7100 over aluminum surfaces. The immersive experimental data concluded the following outcomes.

1 2^{nd} CHF occurred earlier with 6 - 13% less performance rather than 1^{st} CHFs on the calibrated smooth surfaces, while the maximum $HTCs$ of the substrates were united. On the contrary, there was a gradual enhancement in 2^{nd} CHF compared to the initial one on the rough and uncalibrated smooth surfaces. On the other hand, HTC and CHF of the surfaces exposed to continuous boiling in all scenarios showed remarkable enhancements corresponding to the performances just before the operation. The agreement in the heat transfer performances can be ascribed to the unlike oxide layer formations, which inevitably provoke additional bubble growths.

2 The exposed surfaces to continuous boiling processes and boiling crises were imposed to noticeable chemistry and morphology in microscales. The oxide compounds of cuprous (Cu_2O) and cubic oxide (CuO) oxide and copper oxide were highly predicted on the operated surfaces. In most, the compounds growth in distinctive geometries generated nano crevices, pores, and re-entrants, which might be the enhancing factor for nucleation sites. The heterogeneously spread structures raised the surface roughness and wetting conditions partially by generating biphilic surfaces.

3 The rate of heat flux in the continuous boiling operations did not significantly differ the thermal performance, while the CHF*s of the surfaces showed a consistency. However, the shape and density of the copper oxide particles might be diverse on heat flux levels. This variation was certified by the temperature decreases in a continuous process, where the highest temperature drop with 5 K was observed at the highest heat flux of 60 W/cm^2 .

4 After an amount of decrease in 2nd CHF*s, a gradual increase in CHF*s was observed as the number of subsequent runs was increased. The maximum CHF* was on the substrate S8 exposed to four boiling crises. On the other hand, the surface S5, which was not exposed to boiling crises showed the best *HTC* enhancement with 1 W/cm^2 in time during the continuous boiling process with an enormous temperature decrease of 6.4 K. Also, the maximum *HTC*s increased up to critical degrees of 5.11 and 5.42 W/cm^2 , while the number of runs increased. Furthermore, a homogenous oxide layer formed on the surface non-exposed to boiling crises, while a heterogeneous formation existed on the rest surfaces consisting of cubic shaped, leaf-like, and cancellous structures.

5 The surfaces stabilized in heat transfer after a long-term boiling exposure by demonstrating identical heat flux performances at the same wall superheats. Once

the timeline of the constant heat flux application at 45 W/cm^2 was extended, the CHF** doubled relative to the initial performance by increasing up to 144.7 W/cm^2 . Additionally, there was no significant change in HTC during the 2nd consecutive 24 hours boiling operation.

6 The mostly formed cubic shaped micro-particles existed on the rough surfaces (treated with silicon carbide abrasive paper) homogenously but did not have an estimable contribution to heat transfer. It was observed that the densely packed oxide formations can close-up the micro-channels and decreases the surface roughness, which brings a performance degradation. In addition, the peroxidation that emerged in the calibration process did not impact the heat transfer significantly, but surprisingly, 2nd CHF performance increased corresponding to the initial one.

7 As a result of the parametric study, micro-drilled surfaces enhanced BHT performance in most conditions whereas subcooling and pressure are great contributors to nucleate boiling heat transfer. The best performance was 181 W/cm^2 on the surfaces with $200 \text{ }\mu\text{m}$ drills at 1.5 bar and 10 K subcooling liquid temperature, where it's nearly 3 times the bare surface performance at 1 bar and saturation.

8 Performing subcooled boiling increases the wall superheat and CHF at a tremendous rate where HTC had a negative impact. Moreover, the positive effect of subcooling on BHT performance is more visible on a plain surface and MD02-1 which have smaller scratches and artificial microgrooves by micro-drill machining, respectively.

9 Two production techniques, lathe machining (LM) and additive manufacturing (AM), for polished surfaces were compared in terms of heat transfer, and it was concluded that there is no considerable change in HTC , whereas there is a substantial variation in wall temperature at comparable heat fluxes.

10 The boiling performance of the additive manufactured plain surface showed a significant enhancement by using the advantage of surface roughness, which controls the nucleation site density and capillary wicking. In addition to plain surface analysis, AM microchannels improved BHT enormously by separating vapor escape and using a suction-evaporation mechanism. With the advantages of enhanced properties, MC3, which has inverse V-shape channel geometries, showed the best performance with 130% and 386% enhancement compared to AM polished surface on CHF and HTC , respectively. The high-speed images captured at nucleate boiling and just before CHF levels supported the surface performances.

CHAPTER V

RECOMMENDED FUTURE WORK

Significant advancements have been made in comprehending the mechanisms underlying nucleate pool boiling, a research area that has spanned over a century. In recent years, researchers have made notable progress by considering various factors such as manufacturing techniques, material advancements, chemical applications, and a wide range of operating conditions and combinations. Each study contributes uniquely to the overall understanding of phase change phenomena. However, as industrial systems continue to generate increasing amounts of waste heat and system miniaturization becomes integral to design, the demand for complex geometries in heater surfaces and highly efficient working fluids with exceptional thermophysical properties becomes more pronounced. Recent research studies have highlighted the significant improvement in nucleate pool boiling performance, reaching up to 420 W/cm^2 , through the utilization of microscale surface modifications. Microchannel and microporous surfaces have emerged as promising methods for enhancing heat transfer. By employing micro-texturing techniques, additional surface cavities can be generated using selectively manufactured geometries, leading to a higher number of active nucleation sites and improved rewetting ability. This results in an elevation of the heat transfer limit. Furthermore, additive manufacturing processes offer a highly advantageous approach, allowing for precise and flexible micro-structuring on bare surfaces. This technology saves time, facilitates high-precision surface manufacturing, and provides a wide range of options for geometric selection and miniaturization. A very controversial example can be seen in the is the development of the SpaceX Falcon 9 rocket. In earlier iterations, traditional manufacturing techniques were predominantly used,

involving machining and assembly of various components. However, in the latest prototype, additive manufacturing techniques have been employed to create intricate and lightweight parts, such as the rocket engine components. This shift has allowed for more precise control over the design, improved performance, and enhanced efficiency. By utilizing additive manufacturing, the Falcon 9 rocket demonstrates the potential of this technology to revolutionize the manufacturing of complex surfaces and parts for boiling applications. Regarding the choice of working fluids, refrigerants, dielectric liquids, and nanofluids are preferred due to their ability to enhance bubble generation and heat transfer coefficients. However, water still demonstrates satisfactory performance. Additionally, graphene has emerged as an innovative and suitable material for liquid mixtures, leading to exceptional heat fluxes. Graphene nanofluids exhibit their best performance on horizontal surfaces, while surface orientation becomes advantageous for heat transfer rates at lower heat fluxes. The study illustrated that in the future, additive manufacturing technology is expected to play a significant role in producing eccentric geometries in micro-scales. Hence, this opens up new possibilities for designing highly efficient heat transfer systems that can be tailored to specific applications and requirements. The understanding and utilization of nonhomogeneous micro formations in oxide layers on heating metal surfaces are likely to be further explored in the future. Indeed, this extremely critical factor which presumably is not simple to simulate numerically cannot be ignored in the boiling experiments on metallic surfaces and the design of such high-performance thermal systems. To put it in a nutshell, current technologies comprise several different operating principles, varying materials, the apparent change in an operating state, and system complexity. To overcome the resulting technical boundaries for thermal management systems, one has to join multiple techniques and should take into account to ensure maximum efficiency, long-term reliability, and a wide range of flexibility by tracking several novel future perspectives such as mechanical surface enhancement&design at micro-scale

together with a combination of cost-effective highly capable working fluids consisting of nano-sized particles acting as a thermal booster.



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