

Laser Surface Engineering for Boiling Heat Transfer Applications



Matevž Zupančič and Peter Gregorčič

1 Introduction

Global primary energy consumption grew at an average rate of 1.5% per year in the last decade, and the world's total power demand is estimated to increase from 18 TW (in 2018) to over 23 TW by 2040 [1, 2]. Since thermal energy plays a primary role in the world's total energy, the efficiency of thermal energy conversion is expected to be a very important factor in the technological progress of modern society. *Phase change* is considered to be the most effective and technically controllable mechanism of heat transfer due to the large change in enthalpy within a small temperature difference, allowing high energy transfer rates in high heat flux applications. *Liquid vapor phase change* in particular is a part of our everyday life and is utilized for cooling and general heat transfer in many applications on various scales – from boiling water reactors in large nuclear power plants [3] to small heat pipes, which are massively produced and are employed in computers, mobile phones, solar collectors, space applications and other fields [4, 5]. In addition to that, emerging technologies including electric vehicles, energy storage and renewable energy-based power generation systems will also need to consider phase-change heat transfer for cooling purposes [6]. Miniaturization and future development of high heat flux devices in terms of performance and safety concerns, therefore, depend on the enhancements in phase-change heat transfer.

Over the last century, *nucleate boiling* became one of the primary topics in liquid vapor heat transfer research [7, 8] with a constant goal of enhancing the boiling process in terms of minimizing the *surface temperature* and increasing the *critical heat flux* (CHF), which defines the maximum attainable heat flux in the nucleate boiling regime [9–14]. Finding the ultimate enhancement approach is a challenging task, since the boiling process is a complex phenomenon and its definite physical

M. Zupančič · P. Gregorčič (✉)

Faculty of Mechanical Engineering, University of Ljubljana, Ljubljana, Slovenia

e-mail: peter.gregorcic@fs.uni-lj.si

model is still not defined, which means that experimental approach to research is unavoidable.

Nucleate boiling depends on a multitude of factors, which can be divided into the following categories:

- *Surface characteristics*, including geometric and material properties, topography on the micro- and nanometre scale, surface chemistry, transient heat transfer inside the solid material, etc.
- *Fluid properties*, including physical and thermal properties, mixture composition, and impurities
- *The interaction between the surface and the fluid* through wettability and adsorption
- *Operating conditions*, such as surface orientation, pressure, liquid subcooling, gravity, electric and magnetic fields, etc.

For a given set of operating conditions and working fluid, the surface characteristics, including micro- and nanoroughness, wettability, surface chemistry and porosity all have a significant impact on boiling process. The main enhancement approach, therefore, is based on the modification (functionalization) of the boiling surface in order to achieve a lower surface temperature at the onset of boiling, alongside increasing the density of active nucleation sites, limiting the bubble growth and finally delaying the dry-out as much as possible. Surface modification methods, thoroughly summarized in recent reviews [15, 16], mostly include mechanical machining [17–19], sintering [9, 20, 21], electrodeposition [22–26], sputtering [27], oxidation [28, 29], growth of nanowires and nanotubes [30–33], (nanoparticle) coatings [34, 35], micro-/nanoelectromechanical techniques [36–39] or combinations of the above methods [40–45]. Usual drawbacks of the employed techniques are expensive production, mechanical and/or thermal instability of surfaces, problematic scale-up and difficult implementation on real three-dimensional surface geometries. As it will be presented in this chapter, *laser surface engineering* has the potential to overcome the current limitations for further miniaturization of microelectronic devices and to increase performance and safety in high heat flux systems [46].

The surface wettability in general significantly influences the boiling performance. For *ideal* surfaces (i.e. chemically homogeneous, chemically stable and completely flat), this is usually explained by using two limiting cases: superhydrophobic (very poorly wetted) and superhydrophilic (very well wetted) surfaces. Here, the prefix “hydro” is related to liquids in general [47, 48], while the prefix “hydro” is used when we specifically describe the results connected with water. (Super) hydrophobic surfaces prefer to be in contact with the vapor phase rather the liquid phase. This promotes the activation of nucleation, reduces the temperature of the onset of nucleate boiling and increases the heat transfer coefficient at low heat fluxes, *but* decreases the critical heat flux. On the contrary, the (super)hydrophilic surfaces delay the critical heat flux *but* increase the surface superheat at low heat fluxes. From these two limit cases, it clearly follows that surface wettability significantly changes the key boiling heat transfer parameters. However, as it will be

discussed in this chapter, the ideal surfaces practically do not exist in real applications. Thus, the measured boiling performance cannot be sufficiently explained without appropriate consideration of the surface topography. Additionally, the surface chemistry and, consequently, wettability may even change during boiling due to intense processes, including the vaporization of the working fluid, high surface temperatures and high frequency temperature variations due to bubble nucleation [49].

This chapter provides a background in nucleate pool boiling needed to explain the current understating of the effects of surface wettability on boiling heat transfer. Afterwards, it introduces direct laser texturing as an emerging technology to produce surfaces with different wetting properties and morphological features. With an emphasis on the results obtained by the authors, the capabilities of surfaces functionalized by nanosecond laser pulses to enhance saturated nucleate pool boiling with water and other fluids are demonstrated. It is also explained how laser surface engineering can be used to decrease the influence of the surface wettability on the key boiling heat transfer parameters and – in this way – to ensure a more stable heat transfer processes by utilizing functionalized surfaces.

2 Introduction to Nucleate Pool Boiling and the Effects of Surface Wettability

Pool boiling is a process of heat transfer between a heated surface and a seemingly stationary liquid in such a way that generation of vapor occurs. The necessary condition is that the temperature of the surface T_w exceeds the liquid's saturation temperature T_{sat} at the given operating pressure. In normal gravity, the growing bubbles detach from the surface mostly due to buoyancy forces, whereas the surrounding liquid near the surface is in motion due to free convection and periodic bubble growth/detachment. Another mode of boiling is forced convection boiling (also termed as flow boiling), where fluid motion is assisted by external flow. Another classification of boiling is through the temperature of the bulk liquid. In subcooled boiling, the temperature of the liquid is below the saturation temperature and the vapor bubbles typically condense before reaching the free surface. In saturated nucleate pool boiling, which is investigated in this chapter, the entire liquid is under saturated conditions, and the heat transferred from the solid to the liquid can be formalized with the Newton's law of cooling

$$q'' = h(T_w - T_{\text{sat}}) \quad (1)$$

where q'' is the heat flux typically expressed in W cm^{-2} or kW m^{-2} , h stands for the heat transfer coefficient usually given in $\text{kW m}^{-2} \text{K}^{-1}$ and $(T_w - T_{\text{sat}})$ is the wall superheat known also as the surface excess temperature in K.

In 1934, Nukiyama [7] studied boiling regimes through an experiment with Joule-heated, horizontally oriented nichrome and platinum wires in a saturated pool

of water. With further experiments by Drew and Mueller [50], it was possible to plot a so-called boiling curve depicting the relationship between the heat flux and the wall superheat, as presented in Fig. 1. At low heat flux, the natural convection sufficiently removes the entire heat from the surface, and no vapor is generated. In this region, heat is removed from the surface by superheating of the liquid, which subsequently travels away from the surface under the effects of buoyancy. As the wall superheat is increased to a few degrees above T_{sat} , bubbles eventually start to form, which is termed as the onset of nucleate boiling.

On a smooth, clean surface, this event usually causes a temperature drop as the heat transfer coefficient suddenly increases. At the onset of nucleate boiling regime, isolated bubbles periodically form on active nucleation sites and are buoying off the surface. Heat is removed from the surface in the form of latent heat of the phase change and enhanced convective heat transfer due to the agitation of the liquid near the surface. As the wall superheat increases further, nucleation site density and nucleation frequency also increase, causing horizontal and vertical coalescence of bubbles. When the bubble population becomes too high and there is virtually no

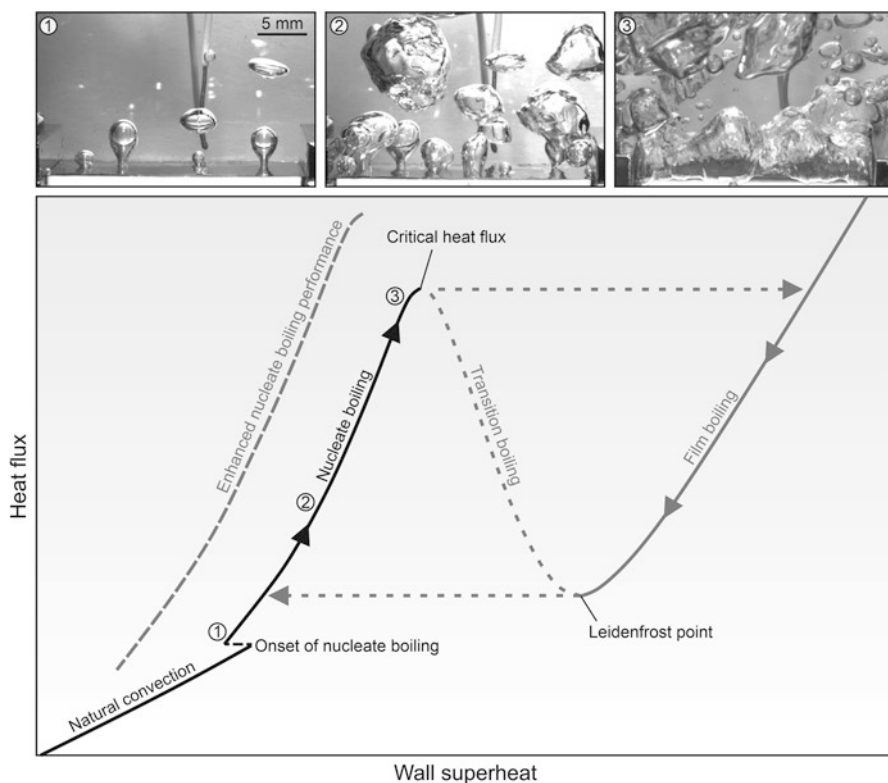


Fig. 1 Typical pool boiling curve (not to scale) and high-speed images at three different heat fluxes (50, 200, and 1150 kW m^{-2}) during saturated pool boiling of water at 1 atm on horizontal Joule-heated stainless steel surface

possibility to create more independent (non-coalescing) nucleation sites on the surface, the insulating vapor blanket entirely covers the surface and significantly decreases heat transfer coefficient. At the moment of reaching this boiling crisis, the heat flux is termed as the critical heat flux (CHF).

In typical boiling heat transfer applications, the heat flux is the independent variable, meaning that by increasing the heat flux just beyond the CHF, the wall temperatures suddenly increases and the film boiling regime ensues. When water is used as the working fluid, this direct transition usually causes the heater to reach its melting point and causes the eventual burnout. However, if the wall superheat is the controlled variable [50] or the surface material can withstand relatively high temperatures (e.g. platinum), the film boiling regime can be sustained. In this regime, the heat is transferred from the surface both by conduction and radiation through the vapor layer on the surface. When the heat flux is lowered again, the boiling curve follows a different path and reaches so-called Leidenfrost point. A further decrease in the heat flux causes a transition back to the nucleate boiling regime, accompanied by a sudden temperature decrease. This loop in a boiling curve is known as the boiling hysteresis and will not be further investigated within this chapter. Clearly, nucleate boiling is the region of interest for most practical applications with the CHF as the upper limit of operation. Hence, this chapter will only consider the nucleate boiling regime.

2.1 Bubble Nucleation

To initiate bubble growth, the primary requirement is the existence of a vapor nucleus inside the liquid. The formation of nuclei inside the superheated liquid resulting from thermal fluctuations of liquid molecules is called *homogeneous nucleation*. When the vapor is formed on the heated solid surface or a suspended nonwetted material, this is known as the *heterogeneous nucleation*. Based on either the thermodynamic or the kinetic limit, the required homogenous nucleation temperature for water at atmospheric pressure is around 300 °C (e.g. 200 K of wall superheat), which is at least one order of magnitude higher than the temperature normally observed in boiling applications. Clearly the surface itself serves as a catalyst and reduces the energy required for nucleation. In practice, heterogeneous nucleation initiates vapor formation, whereas the wettability and morphology of the surface play a key role.

When considering bubble formation on a completely smooth horizontal surface (Fig. 2a), the homogenous nucleation temperature is reduced by a factor $f(\theta)$ as proposed by Cole [51]:

$$f(\theta) = \sqrt{\frac{1}{4}(2 + 3\cos\theta - \cos^3\theta)} \quad (2)$$

where θ is the static contact angle of liquid. When liquid completely wets the surface ($\theta = 0$), the $f(\theta)$ equals 1 and the required nucleation temperature is the same as

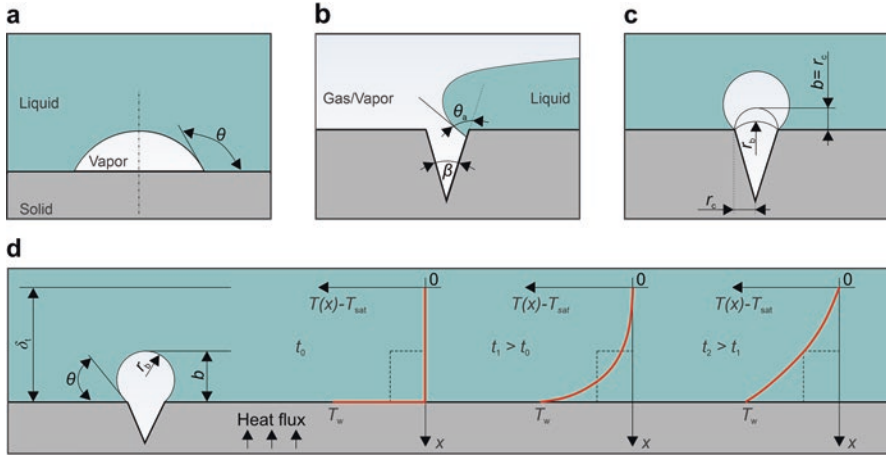


Fig. 2 (a) Bubble embryo on an ideal smooth surface; (b) trapping of gas/vapor inside a conical cavity; (c) presentation of the critical hemispherical condition; and (d) model of Hsu's nucleation criterion

for the homogenous nucleation. In the ideal nonwetting case (when $\theta = 180^\circ$), the $f(\theta)$ equals 0, and nucleation will occur at no superheat of the surface. Here, two facts should be pointed out:

- A completely smooth surface with ideal nonwetting behaviour has never been recorded and cannot be fabricated for practical use
- The reduction of temperature proposed by the $f(\theta)$ factor is not sufficient to explain the actual nucleation temperatures recorded in experiments.

The reason that the actual nucleation temperatures are lower than predicted is the existence of cavities on the heated surface. Naturally present or artificially produced cavities might trap vapor or gas that act as a nucleation embryo. If a certain cavity remains filled with vapor (also due to vapor spreading from neighbouring bubbles) and continues to be active once the nucleation is initiated, it is defined as an *active nucleation site*.

To initially entrap a gas inside the conical cavity, Bankoff [52] proposed that advancing contact angle θ_a must be larger than the groove angle β (Fig. 2b). In this situation, the liquid hits the opposite cavity wall before reaching the bottom, therefore entrapping a certain amount of gas or vapor. As the bubble grows from a cavity with a mouth radius r_c , it passes through the critical hemispherical condition [53], where the bubble tip is at the same distance from the surface as the cavity radius value (Fig. 2c). Here, the bubble radius is at its smallest, and the pressure inside is at its maximum as predicted by the Young-Laplace equation. In other words, the maximum amount of superheat is needed at this point to keep the bubble growing. For low-surface-tension fluids, however, the initial radius of a gas bubble inside the cavity might be smaller than the cavity radius. In this case, the wall superheat is larger than the value predicted by the critical hemispherical condition.

Analysis in the previous paragraph assumes a uniform and equal temperature of the liquid, the vapor and the wall. In practical boiling, however, the heat is transferred from a heated surface to the cooler liquid. When a bubble grows inside the liquid, it moves through the thermal boundary layer adjacent to the wall, and the criteria specified for uniform temperature are not necessarily sufficient. Experiments have shown that for a certain wall superheat, there is a certain size range of active nucleation cavities. This was taken into account by Hsu [54], who derived a nucleation criterion that later became one of the most commonly used in the pool boiling community. Let us assume a small bubble nucleus of a radius r_b sitting at the mouth of the vapor trapping cavity (Fig. 2d). The nucleus is leftover vapor from the previous bubble departure. At time t_0 , the entire liquid is in saturated conditions, and thermal boundary layer starts to form again following the previous bubble growth and departure cycle. With time, the liquid is heated up inside the boundary layer δ_t due to heat transfer from the hotter surface. Only when the liquid temperature reaches the saturation temperature of the vapor inside the bubble (which can be calculated from the Clausius-Clapeyron equation) over the entire bubble height at time t_2 , the bubble starts to grow. By accounting for all geometrical constraints and the transient temperature distribution inside the thermal boundary layer from Carslaw and Jaeger [55], Hsu derived the final equation to determine the size range of possibly active cavities

$$\{r_{c,\min}, r_{c,\max}\} = \frac{\delta_t \sin \theta}{2(1 + \cos \theta)} \left(1 \mp \sqrt{1 - \frac{8\gamma_{LV} T_{\text{sat}} (1 + \cos \theta)}{h_{LV} \rho_V \delta_t (T_w - T_{\text{sat}})}} \right) \quad (3)$$

where γ_{LV} is the liquid vapor surface tension, h_{LV} is the heat of vaporization and ρ_V is the vapor density. Figure 3 shows three different ranges (for three different wettabilities) of active cavity size versus wall superheat for saturated boiling of water at 1 atm. It is obvious that superhydrophobic surfaces provide the lowest needed superheat for the onset of boiling and allow nucleation from cavities with a radius from roughly 1 μm up to 1000 μm (pink region in Fig. 3). On superhydrophilic surfaces, however, the cavities should be significantly smaller, and the size range of possibly active cavities narrows down to about 0.2–10 μm (blue region in Fig. 3). It should be pointed out that the overlapping of different regions in Fig. 3 clearly shows that there exists a certain range of cavity size that are suitable as active nucleation sites for surfaces with different contact angles.

In the above analysis, it should be noted that the thermal boundary layer constantly changes during boiling and the optimal cavity size range is therefore subjected to perpetual change as well. Moreover, Hsu's model considers the static contact angle of the liquid, even though the receding contact angle is the one that is observed during a bubble growth. This was later addressed by Kandlikar et al. [56], who provided a modification of the criterion from Eq. (3) specifically for flow boiling processes [57–59]. Bubble nucleation is obviously a very complex phenomenon, and it would be impossible to even list the existing publications and their

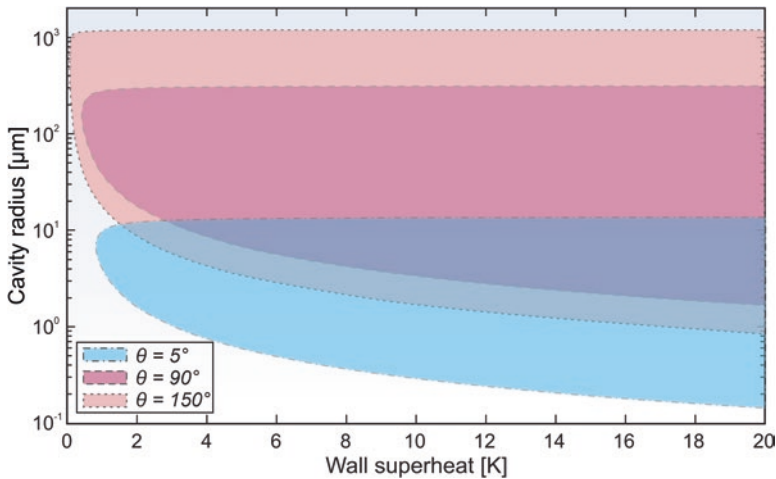


Fig. 3 Effective cavity radius range versus wall superheat for saturated boiling of water at 1 atm at three different contact angles. The calculation is based on the Hsu's nucleation criterion (Eq. (12.3)), while the thermal boundary layer thickness δ_t was assumed as the ratio between the thermal conductivity of the water ($0.679 \text{ W m}^{-1} \text{ K}^{-1}$) and selected value of the heat transfer coefficient for natural convection ($2.2 \text{ kW m}^{-2} \text{ K}^{-1}$)

findings within a single chapter. Instead, we will provide some key points that are also necessary for the further discussion of the experimental results:

- To initiate the boiling process at reasonably low superheats (e.g. up to 5 K), the surface must contain gas or vapor trapping cavities. The general rule is that poorly wetted surface will allow vapor trapping within steep and shallow cavities [51], whereas the well wetted surface will only allow trapping within steep cavities. The combination of a steep cavity and well wetted surface, however, makes it difficult to entrap vapor again after the cavity was completely filled with liquid. Hence, poorly wetted cavities have the greatest tendency to entrap gas or vapor and show the best potential to initiate boiling process.
- The boiling onset and the initial population of active nucleation sites depend greatly on the concentration of non-condensable gasses inside the liquid. This is especially important for poorly wetted surfaces. Furthermore, the nucleation process from non-condensable gas embryo (e.g. air) in comparison to vapor embryo is very different [52, 60, 61], which will be discussed later.
- Surfaces with strictly defined cavity size might only be optimal to provide nucleation under certain operating conditions. On the other hand, surfaces that incorporate cavities in a wide range of sizes might allow effective nucleation within the entire nucleate boiling regime (e.g. across a wide range of superheats), for different wettability states [62] and different types of fluids [46].
- Over two decades ago [63], atomic force microscopy (AFM) revealed the existence of so-called surface nanobubbles – spherical caps of gas (possibly even vapor) sitting on top of a poorly wetted surface. Observed nanobubbles with

diameters up to 1 μm and height up to several tens of nm exhibit extremely high contact angles in water (over 150° measured through the water), which makes it possible for them to become stable over a longer period [64, 65]. It was proved that nanobubbles can cause a relatively low temperature at the onset of nucleate boiling even on a nanoscopically smooth surface [66], which cannot be explained through existing heterogeneous nucleation models. In the near future, the emerging fluorescence microscopy techniques [67–69] with submicron spatial resolution might be able to provide a completely new understanding of the mechanisms behind bubble nucleation.

2.2 Bubble Growth and Departure

Once the conditions to initiate a nucleation event are fulfilled and the bubble starts to grow, the bubble growth time t_g and waiting time t_w can be defined by analysing the surface temperature at the nucleation site and the bubble footprint radius r_f (Fig. 4). Start of the nucleation is observed as a sharp temperature drop with a high local heat flux [70, 71] where both the bubble radius r_b and the bubble footprint radius increase. After the buoyancy force starts to lift the bubble off the surface, the footprint decreases until finally the bubble is detached. The period between the start of nucleation and detachment is known as the bubble growth time t_g . After the bubble departure, cold liquid rewets the nucleation site and a thermal boundary layer starts to form again until the next nucleation occurs. This time is termed as the

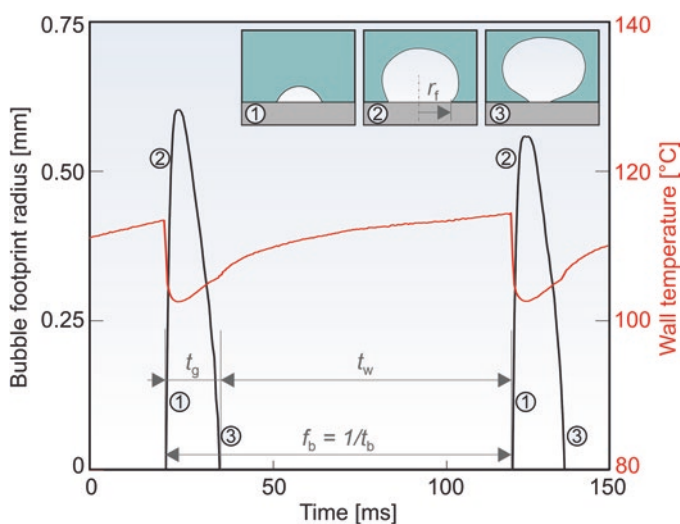


Fig. 4 Typical wall temperature and the bubble's footprint radius development during the bubble life cycle in the saturated pool boiling of water on smooth stainless steel at 50 kW m^{-2}

waiting time t_w . The period t_b is the sum of the growth and the waiting time, and the nucleation frequency $f_b = 1/t_b$ is inversely proportional to it.

As the heat flux is increased towards the CHF or if the nucleation requires relatively large superheats that result in very large bubbles, there is a chance that the liquid microlayer beneath the bubble dries out [72–74]. In this case, the local heat flux underneath the bubble is decreased dramatically followed by the formation of a local hot spot. In order to keep the entire boiling surface at a low temperature and to avoid the burnout of the heater, the hot spot formation should be avoided or delayed as much as possible.

Fritz [8] was the first who correlated the bubble departure radius r_d and contact angle as a result of balancing the buoyancy with the surface tension force, as schematically presented in Fig. 5a:

$$r_d = 0.0104\theta \sqrt{\frac{\gamma_{LV}}{g(\rho_L - \rho_V)}} \quad (4)$$

where g stands for the amplitude of the gravitational acceleration and θ is the static contact angle in degrees. This correlation suggests that bubble departure diameter linearly increases with the contact angle. However, Phan et al. [75] analysed the situation from another perspective (Fig. 5b). When considering two ideally spherical bubbles with the same footprint and different contact angles, the one with a larger contact angle will always be bigger due to simple geometric reasons. They

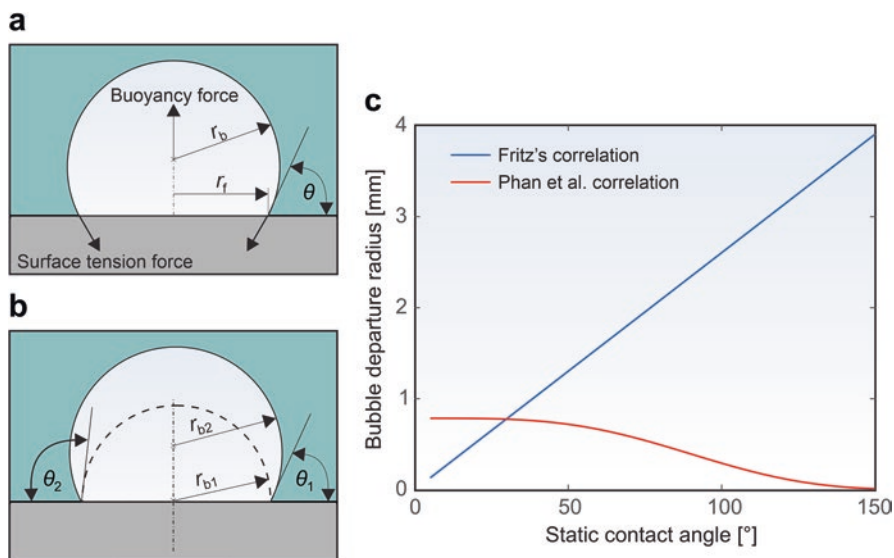


Fig. 5 (a) Illustration of buoyancy and surface tension forces acting on a growing bubble; (b) effect of the contact angle on the size of a spherical bubble; and (c) comparison of two correlations (Eq. (12.4), the blue curve, and Eq. (12.5), the red curve) for the bubble detachment radius for saturated pool boiling of water at 1 atm

modified the Fritz's model by taking into account the energy factor $f(\theta)$ needed to form a bubble from Eq. (2). In this way, they developed a new correlation for the bubble departure radius:

$$r_d = 0.3135 \frac{(2 + 3 \cos \theta - \cos^3 \theta)}{4} \sqrt{\frac{\gamma_{LV}}{g(\rho_L - \rho_V)}} \quad (5)$$

From Fig. 5c, it follows that the correlations by Eqs. (4) and (5) provide completely different trends for the bubble departure radius as a function of the static contact angle. However, the experimental results on surfaces with specific wettability, roughness, fluid type and operating conditions have shown that both correlations can be valid under certain conditions. This indicates that all of these factors play an important role in bubble growth, and despite numerous investigations, there is still no uniform model to accurately predict the bubble size and its growth time for a variety of applications. Even so, one can summarize the most important conclusions, which are mostly based on the experimental results:

- Investigators usually observe a decrease in the bubble departure diameter when a surface is engineered in such a way that lowers its static contact angle [34, 76–78]. On a well wetted surface, the adhesion of the liquid limits the bubble growth, which in turn results in smaller bubbles and higher nucleation frequency assuming the nucleation site density is kept constant. Looking from another perspective, poorly wetted surface will produce larger bubbles and provide longer growth times [79–81], which increases the possibility of formation of undesired local dry-outs and hot spots.
- While maintaining the same pressure and gravity, a decrease in bubble diameter can also be achieved by lowering the fluid's surface tension, which can be achieved either by using a different type of fluid [46, 82], by adding an additional miscible fluid with low surface tension [83–85] or by adding nanoparticles [86]. These techniques can be used to decrease the bubble size, even though this does not necessarily result in an enhanced heat transfer coefficient as the required wall superheat for the bubble nucleation might also increase.
- Discussion about the effects of wettability on the bubble growth is often limited to the static contact angle, as the difference in advancing and receding contact angle in pool boiling is within only $\pm 5^\circ$ based on the results from Ramanujapu, Dhir [87]. Recent findings [76], however, proved that some types of poorly wetted surfaces can exhibit low receding contact angles, which allows low temperature at the onset of boiling and – at the same time – prevents vapor spreading. This enhances the heat transfer coefficient and does not decrease the critical heat flux, which will be discussed in Sect. 4.4.
- Bubble size and bubble dynamics depend not only on wettability, surface roughness and fluid properties, but are influenced also by temperature fields in the solid and the liquid, contribution of microlayer evaporation and complex interactions with neighbouring bubbles. Therefore, tailoring the surface to provide the highest possible nucleation frequency and the nucleation site density does not

necessarily result in the best boiling performance. Distribution of active nucleation sites has to be considered as well, which will be partially discussed in the next section.

2.3 *Pool Boiling Enhancement*

Topographical features and wettability of the boiling surface play an important role in the nucleation, growth and departure of vapor bubbles. Therefore, specific surface characteristics can be used to enhance the heat transfer coefficient (HTC) and the critical heat flux (CHF). Increasing the surface wettability generally increases the CHF but also increases the activation energy for bubble nucleation, which leads to decreased HTC. On the other hand, increasing the nucleation site density and lowering the bubble activation temperature by decreasing the surface wettability and providing appropriate nucleation cavities will increase the HTC, while the CHF is typically decreased on such surfaces. One of the best approaches to enhance both the HTC and the CHF is by locally altering the surface features and chemistry to achieve controlled nucleation, low onset of boiling, effective removal of bubbles and fast liquid replenishment in such a way that formation of large vapor patches is delayed and average surface temperature is maintained as low as possible.

Despite several decades of research, there is still no universal model to accurately predict the HTC and CHF for the wide spectrum of possible boiling conditions [88, 89]. The early hydrodynamic model [90] suggest that CHF value in saturated pool boiling of water is around 1.2 MW m^{-2} . The later developed force-balance model [91] that also includes the effects of the contact angle predicts a decrease in CHF with increasing contact angle. Both of the aforementioned models somewhat accurately predict the measured CHF values on smooth untreated surfaces, which are in the order of $0.6\text{--}1.4 \text{ MW m}^{-2}$ at about $17\text{--}35 \text{ K}$ of superheat (Fig. 6a). The maximum reported HTC values on untreated surfaces are usually in the range of $20\text{--}60 \text{ kW m}^{-2} \text{ K}^{-1}$. Using water as the working fluid, the CHF alone can be enhanced by coupling the hydrophilicity and micro-/nanoroughness via nanowires or nanotubes [31, 92, 93], bio-templating [12], sintering [9, 20, 21], hierarchical structuring [42, 94, 95] or oxidation [28]. These kinds of surfaces can provide enhanced capillary force or wicking [96] to effectively rewet the surface and push the CHF to about $2\text{--}3 \text{ MW m}^{-2}$ with generally limited enhancement in HTC.

To answer the need for simultaneous enhancement of HTC and CHF, researchers developed different surface engineering approaches that include heterogeneous surface chemistry and geometry from macro- to nanoscale in order to gain control over the bubble nucleation. One of the most straightforward techniques shown in Fig. 6b was introduced by Rahman et al. [97], who fabricated alternating regions with low and high thermal conductivity on a copper surface. Heat is conducted mostly through the copper, which represents the active boiling surface, while the insulating epoxy is cooler, thus preventing bubble nucleation and providing a downward pathway for the liquid. Despite the decrease in active boiling area compared to untreated

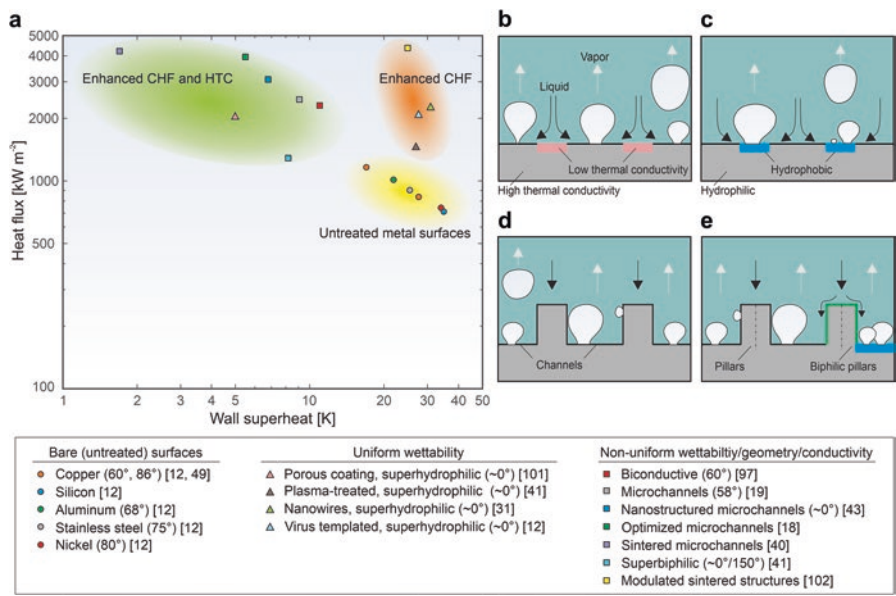


Fig. 6 (a) CHF values recorded on bare metal surfaces and various engineered surfaces reported in the literature [12, 18, 19, 31, 40, 41, 43, 49, 97, 101, 102], achieved under atmospheric saturated pool boiling of water. Where applicable, values in round brackets in the legend represent the reported static contact angles of water. Schematic depiction of how boiling enhancement is achieved by the spatial control over bubble nucleation on (b) biconductive surfaces, (c) biphilic surfaces, (d) open mini-/microchannels and (e) mini-/micropillars

copper, the fivefold enhancement of the HTC ($210 \text{ kW m}^{-2} \text{ K}^{-1}$) and twofold enhancement of the CHF (2.3 MW m^{-2}) was demonstrated on such biconductive surfaces. The authors also show that the optimal pitch of the low-conductivity array is close to the capillary length of water (2.5 mm). Similar control over the nucleation can be achieved on surfaces with mixed hydrophilic and hydrophobic regions (so-called biphilic surfaces), which are shown in Fig. 6c. Surfaces with hydrophobic islands and a hydrophilic background have shown the best enhancement in CHF (up to 1.9 MW m^{-2}), while superbiphilic surfaces demonstrated the HTC of more than $200 \text{ kW m}^{-2} \text{ K}^{-1}$ [41, 98]. In this case, the hydrophobic regions promote bubble nucleation, while the surrounding hydrophilic areas prevent the expansion of the vapor and drive the liquid towards nucleation sites [34].

Effective separation of the vapor and the liquid can also be achieved with mini-/microchannels or pillars (Fig. 6d, e). The bottom of the channel is closer to the heating source and thus hotter making it favourable for nucleation, while the top structures are cooler and less likely to promote bubble formation. This effect can also be enhanced with hydrophobic/hydrophilic patterning. Open microchannel structures with relatively smooth surface demonstrated the CHF of more than 2.4 MW m^{-2} and HTC of $270 \text{ kW m}^{-2} \text{ K}^{-1}$ [19], while the sintered channels were able to dissipate 4.2 MW m^{-2} at the superheat of only 1.7 K ($\text{HTC} > 2400 \text{ kW m}^{-2} \text{ K}^{-1}$) [40]. The

characteristic length scales of optimal surface geometries for biconductive, biphilic or channeled surfaces span from several millimetres down to the micrometre range, indicating that several complex mechanisms are governing the boiling enhancement. This is likely the reason why the universal model of the boiling process is still not defined. Despite great efforts to control and enhance nucleate boiling, the reported CHF values are still far below the kinetic evaporation limit [99, 100], which remains the ultimate goal for future generations.

Currently available techniques for surface engineering provide numerous possibilities of their combinations to develop hybrid surfaces with even better performances. Unfortunately, this usually sacrifices the scalability and increases the complexity as well as the production costs. From this perspective, it seems that further steps should focus on simplified approaches that still have the potential to result in significantly enhanced nucleate boiling performance. We believe that *laser surface engineering* is a promising alternative approach for functionalization of surfaces for controlled and enhanced nucleate boiling, offering both scalability and environmental friendliness. Thus, the next section introduces laser surface engineering and briefly describes how it can be used for the modification of surface topography, chemistry and, consequently, wettability.

3 Laser Surface Engineering

Due to intrinsic properties of laser radiation, its interaction with solid material can induce topographical and chemical modifications on the micrometre and nanometre scale [103–106]. A number of studies have shown that this can be efficiently done using femtosecond [107–110], picosecond [111–113], and nanosecond [114–117] laser pulses as well as continuous-wave laser radiation [118]. These solid surface modifications depend on several parameters, such as light polarization, wavelength, pulse duration, average power, pulse fluence and pulse overlap.

To tailor the functionalized surfaces for specific applications, a new research field, known as *laser surface engineering* [119], emerged in last decade. In general, it comprises the following steps:

- Designing surfaces for various applications
- Modelling and choosing of the laser-processing parameters
- Modifying the surface properties by laser radiation
- Characterizing the fabricated surfaces using different analytical methods
- Testing the engineered surfaces in proposed applications.

The laser surface engineering represents a flexible, scalable and chemical-free method. Thus, it opens up completely new possibilities for the development of new advanced materials that are important for a wide range of engineering applications. We will limit this chapter to engineering of metal surfaces with multiscale μ -cavities as well as variable and/or unstable wettability for boiling heat transfer [46, 49, 62] by using nanosecond laser texturing at pulse fluences significantly exceeding the

threshold fluence for laser ablation [104]. However, this section also briefly presents a general concept of changing the surface wettability through the morphology and chemistry modification by laser-surface interaction. This concept is – in addition to the boiling heat transfer – important also for biomedical [106, 120–122], corrosion [123–126], tribological [127–130] and fluid-flow [131] applications.

3.1 Laser Texturing of Two-Dimensional Surfaces

For laser texturing, a laser system is needed. It consists of (i) a laser source, (ii) focusing optics, (iii) a scanning head, (iv) a controller and (v) the graphical user interface. The laser source is characterized by the wavelength λ given in nm, pulse duration t_p , average power P_{avg} , repetition rate f and the beam quality factor $M^2 > 1$ defining how close is the laser-beam profile to the diffraction-limited beam, also called the Gaussian beam. Pulsed laser sources irradiate light in optical pulses, where the pulse duration t_p goes from several femtoseconds to several milliseconds. In this chapter, we will limit ourselves to laser texturing by nanosecond laser pulses.

A laser pulse is a “package” of a certain number of photons, each carrying the energy of $E_0 = hc/\lambda$, where Planck constant equals $h = 4.14 \times 10^{-15}$ eVs, while the speed of light in vacuum is $c \approx 3 \times 10^8$ m s⁻¹. Thus, most commonly used lasers radiate photons with energies in the range 0.1–10 eV. Not only the photon energy but also the absorption of the laser light in a certain material depends on the wavelength. The pulse energy equals the number of photons, irradiated within a single laser pulse, multiplied by the photon energy. Laser pulses are separated by a time interval, t_0 (Fig. 7). The pulse repetition rate is defined as $f = 1/t_0$ and typically goes from a single pulse to several MHz.

Power is the measure for the temporal flow of the energy. Thus, the average power of the laser radiation is defined as the product of the number of pulses N_p , and the pulse energy E_p divided by time t , needed to irradiate this number of pulses. Since the ratio N_p/t equals the repetition rate, the average power is simply

$$P_{\text{avg}} = E_p f \quad (6)$$

However, the pulse power is changing during the pulse, i.e. it goes from zero to peak power P_p and back to zero (the blue curve in Fig. 7). By using a square approximation, we can define the peak power P_p as

$$P_p = E_p / t_p \quad (7)$$

where the pulse duration t_p is usually measured at full width at half maximum (FWHM) of the pulse-power curve (Fig. 7). If we assume that the pulse power can be described by a Gaussian beam, the peak power, estimated by the square approximation (Eq. (7)), is $1/2\sqrt{\pi/\ln(2)} = 1.06$ times higher than the real peak power (the dashed violet square in Fig. 7).

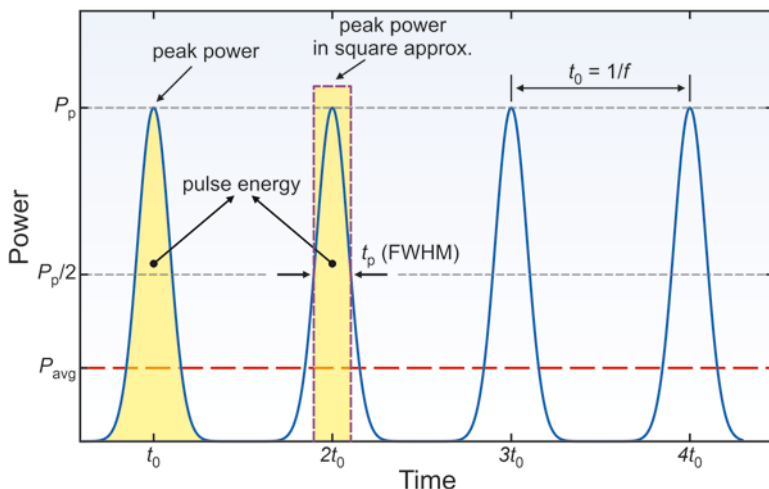


Fig. 7 Schematic presentation of pulse power as a function of time (the blue curve) for the pulse duration of t_p and the pulse repetition rate $1/t_0$. The pulse energy E_p equals the yellow area. The square approximation (with the same pulse energy) is shown by the dashed violet curve, while the dashed red line shows the value of average power

The *focusing optics* is an important component of the laser system, since it defines the area of the beam spot on the processed surface and, consequently, the fluence F given in J cm^{-2} or intensity I in W cm^{-2} . In general, the focusing optics of a laser system consists of a beam expander and a focusing (F-theta or telecentric) lens. The beam expander is usually used to increase the beam diameter D on the focusing lens. This decreases the beam radius in the focus of the lens, called also the beam waist radius w_0 , since the following relation is valid

$$w_0 = \frac{2f_L \lambda}{\pi D} M^2 \quad (8)$$

where f_L stands for the focal length of the lens. It can be observed that the beam quality factor, which is an inherent property of the laser source, significantly influences the smallest attainable beam waist radius. High beam quality is one of the most important advantages of the fibre lasers versus other types of lasers [132].

The *scanning head* usually consists of two mirrors that lead the laser pulses over a two-dimensional surface. Instead of using a scanner, the sample can also be moved using an appropriate x-y positioning system. Both approaches allow processing of the sample in two dimensions. Here, the following three processing parameters are important: the scanning speed v , the scanning line separation Δy and the scanning strategy. The combination of the scanning speed and the repetition rate defines the distance between the centres of consecutive pulses (Fig. 8a):

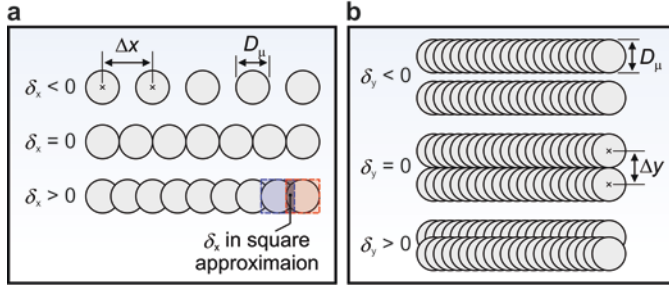


Fig. 8 (a) Overlapping between pulses (in the x direction). The square approximation is schematically presented. (b) Overlapping between μ -channels (in the y direction)

$$\Delta x = \frac{v}{f} \quad (9)$$

Consequently, the pulse overlap (Fig. 8a) is defined as

$$\delta_x = 1 - \frac{\Delta x}{2D_\mu} \quad (10)$$

where D_μ stands for the diameter of the hole (damage) ablated by a single pulse. It should be noted that Eq. (10) is valid for a square approximation (see the last two pulses for $\delta_x > 0$ in Fig. 8a). Some authors use the beam diameter on the surface w instead of D_μ in Eq. (10). However, in this case, this is not the overlap of the laser-induced craters but is instead related to the overlapping of the laser pulses. As we will show later, the crater diameter is not necessarily equal to the beam diameter (in general, $D_\mu \neq w$). When $\delta_x > 0$, the craters overlap and form a micro(μ)-channel, while $\delta_x < 0$ leads to separate μ -craters, as sketched in Fig. 8a.

To process the whole two-dimensional surface, multiple μ -channels need to be fabricated across the surface. Here, the scanning line separation Δy is another important parameter. Like the overlap of consecutive pulses, the overlap of the consecutive lines (Fig. 8b) can be defined as

$$\delta_y = 1 - \frac{\Delta y}{2D_\mu} \quad (11)$$

In Eq. (11), D_μ stands for the width of individual μ -channels.

The two-dimensional surface can be textured in many different ways. The most common *scanning strategies* are schematically shown in Fig. 9 and can be used to control the surface wettability for different applications:

- *0° texturing with equidistant spacing* (Fig. 9a) represents the simplest scanning strategy. Therefore, it was used in plenty of experiments, including the production of laser-textured hydrophobic surfaces for chemical sensing applications

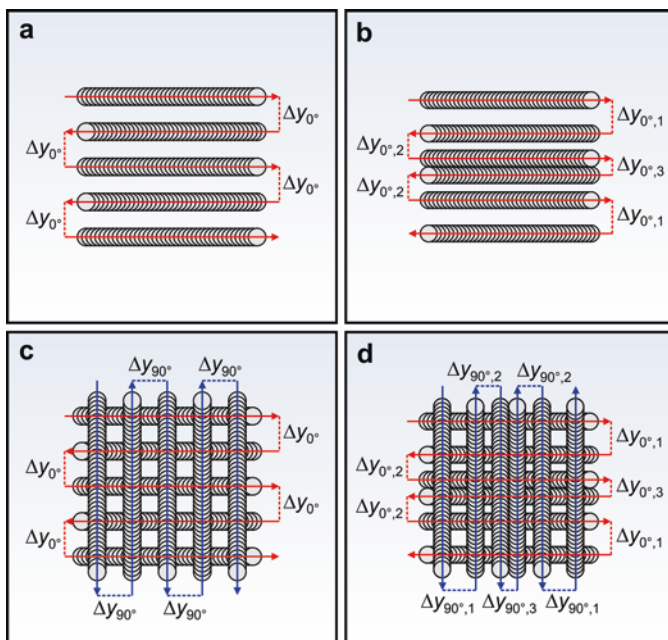


Fig. 9 Schematics of the most common scanning strategies. (a) 0° texturing with equidistant spacing, (b) 0° texturing with variable spacing, (c) $0^\circ/90^\circ$ texturing with equidistant spacing and (d) $0^\circ/90^\circ$ texturing with variable spacing. The direction of the scanning passes is shown by the arrows, and the laser is switched off when leading at the dashed lines

[114], laser texturing of wettability gradients [133], modifying the surface wettability for friction control [128] and the development of biodegradable materials by producing hydrophilic FeMn surfaces that trigger self-driven corrosion [121].

- 0° texturing with variable spacing (Fig. 9b) is a modification of the previous scanning strategy leading to multiscale μ -cavities. Gregorčič et al. [46] used this approach to enhance boiling heat transfer for a diverse range of fluids, independent on their polarity or surface tension, while Zakšek et al. [84] have shown that this approach enhances boiling parameters also for binary mixtures.
- $0^\circ/90^\circ$ texturing with equidistance spacing (Fig. 9c) is the combination of the simplest scanning strategy (0° texturing) in two perpendicular directions—the surface is textured first in x and then in y direction. Trdan et al. [123] used this scanning strategy to study the influence of superhydrophilic-to-superhydrophobic transition on corrosion resistance of laser textured stainless steel. They have shown that this strategy leads to open μ -channels along the last laser scanning direction (90°) and partially closed μ -channels in the perpendicular direction (0°) enabling faster fluid motion in the last scanning direction (90°).
- $0^\circ/90^\circ$ texturing with variable spacing (Fig. 9d) is a modification of the previous scanning strategy. Zupančič et al. [62] used this strategy to produce surfaces with multiscale μ -cavities and non-uniform wettability.

In many cases [111, 117, 134], the surface modification does not depend only on the pulse fluence but also on the total energy irradiated to the surface area, F_{tot}

$$F_{\text{tot}} = N_A \frac{P}{v\Delta y} \quad (12)$$

where the integer N_A tells how many times the whole area is processed (the number of the repetitions across the whole processed area). In case of a single repetition of 0° texturing, $N_A = 1$, while for a single repetition of the $0^\circ/90^\circ$ texturing, $N_A = 2$. If the same surface is processed twice using the $0^\circ/90^\circ$ strategy, $N_A = 4$.

3.2 Influence of Laser Fluence on Surface Topography

It is widely known that laser ablation leads to topographical and chemical modifications of the surface [103–105, 115]. Since the particular change of the surface properties significantly depends on the intensity and/or fluence of laser radiation, laser texturing of metals in an oxygen-containing atmosphere can be classified into the following three regimes:

- *The regime of very low fluences* (i.e. below the threshold fluence for surface ablation), where the oxidation of the metallic surface represents the main modification during the processing. In this case, the topographical modifications of surface on the micrometre level almost disappear and the chemical modification manifests itself the formation of ten or hundred nanometre thick oxide layer that is visible as colorized metal [117, 135].
- *The regime of fluences near the ablation threshold* by polarized laser pulses. In this case, the so-called laser-induced periodic surface structures (LIPSS) [136] appear on the surface as a regular ripple structure with a period scaling from well below to well above the laser wavelength, while its orientation and shape depend on the pulse polarization [111, 137]. LIPSS can be formed using various pulse durations, including femtosecond [138–141], picosecond [111, 142] and nanosecond pulses [116, 143] as well as continuous-wave radiation [144].
- *The regime of high fluences* resulting in ablation and melting of the material. Here, the overlap of craters forms μ -channels, as visible on SEM micrographs in Fig. 10a and a hierarchical structure appears due to the melting and oxidation of the material [104, 106]. Additionally, the remolten material is deposited around the textured μ -channel.

This chapter considers only the laser texturing in the high fluence regime and in the air atmosphere. Figure 10a shows μ -channels on a stainless steel surface induced by a Nd:YAG laser ($\lambda = 1064$ nm) radiating 30 ns long pulses. The sample was textured in the x direction (0° texturing) in the focus. The scanning line separation was constant ($\Delta y = 50$ mm), while the peak fluence was varied in the range of $4.51\text{--}25.1$ J cm $^{-2}$ by changing the average power at a constant repetition rate. The

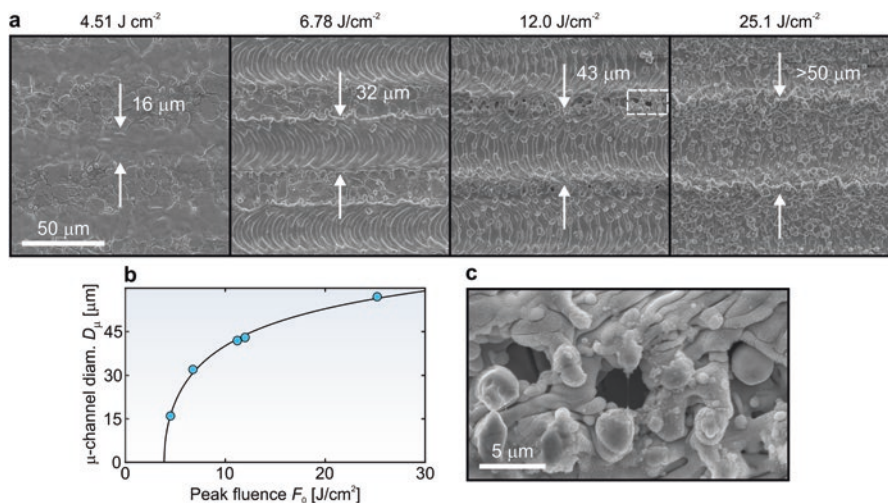


Fig. 10 SEM micrographs of μ -channels at different peak fluences F_0 (shown on the top of each micrograph); scanning line separation in all cases equals $\Delta y = 50 \mu\text{m}$. **(b)** Experimental (points) and theoretical (line; see Eq. (17)) dependence between the μ -channel diameter and the peak fluence F_0 . **(c)** Magnification of the selected area in **(a)** showing the appearance of μ -cavities. (Reproduced and adapted from Ref. [134]. Creative Commons Attribution 4.0 International License)

scanning speed of 150 mm s^{-1} and the repetition rate of 25 kHz resulted in the distance between the consecutive pulses of $\Delta x = 6 \mu\text{m}$, while the beam diameter equalled $w_0 = 29 \mu\text{m}$. Considering $D_\mu = w_0$ in Eq. (9), the overlap of laser pulses equalled $\delta_x = 0.90$. However, it is clearly visible that the μ -channel diameter D_μ depends on the pulse fluence and – for the given processing parameters – goes from 16 to 56 μm . If the real μ -channel diameter is taken into account, Eq. (10) gives that the overlap δ_x changes in the range 0.81–0.95.

The dependence between the μ -channel diameter and the laser fluence can be described by considering a Gaussian beam profile [134], where the fluence $F(z, r)$ is a function of the axial distance from the beam's focus z and radius r is given by

$$F(z, r) = F_0 \frac{w_0^2}{w^2(z)} \exp\left(-2 \frac{r^2}{w^2(z)}\right). \quad (13)$$

In Eq. (13), F_0 stands for the peak fluence at $r = 0$ and $z = 0$, w_0 is the $1/e^2$ beam waist radius in the focus (at $z = 0$) and the beam radius as a function of z is defined as

$$w(z) = w_0 \sqrt{1 + z^2 / z_R^2}. \quad (14)$$

Here, the Rayleigh length, i.e. the distance between the focus and the position z_R , where the area within the beam diameter is doubled, is calculated as

$$z_R = \frac{\pi w_0^2}{\lambda}. \quad (15)$$

Equation (13) can be used to predict the radius of the ablated area $R_\mu = D_\mu/2$ on a surface placed at z , if the following assumptions are made:

- The region of the irradiated surface, where $F(z, r) \geq F_{th}$ is damaged (textured).
- The region with $F(z, r) < F_{th}$ is not damaged (not textured).

Under these assumptions, the radius R_μ of the damaged (textured) surface equals the radius r , where $F(z, r = R_\mu) = F_{th}$. Putting this condition into Eq. (13) gives

$$R_\mu = w_0 \sqrt{\frac{1 + (z/z_R)^2}{2} \ln \left(\frac{F_0 / F_{th}}{1 + (z/z_R)^2} \right)}. \quad (16)$$

Figure 11 shows how the radius of the ablated area changes, when the surface is moved out of the focal plane for different ratios between the peak fluence and the fluence for the ablation threshold F_0/F_{th} . Note that the dimensionless radius R_μ/w_0 is given in the y axis and that the dimensionless distance $|z|/z_R$ is shown in the x axis. It is evident that the radius of the μ -structure, textured in the focal plane, equals the beam waist radius ($R_\mu/w_0 = 1$) only for the ratio $F_0/F_{th} = e^2$. For the ratios $F_0/F_{th} < 3$, the μ -structure radius is *always smaller* than the beam waist radius, and it *decreases* when the surface is placed out of the focal position. However, for $F_0/F_{th} > e^2$, the μ -structure radius is *larger* than the beam waist radius, and it first *increases* by moving the surface out of the focal position. However, if the surface is further moved out of the focus, the R_μ starts to decrease until surface damage is no longer observable.

If the surface is processed in the focus ($z = 0$), Eq. (16) is simplified to

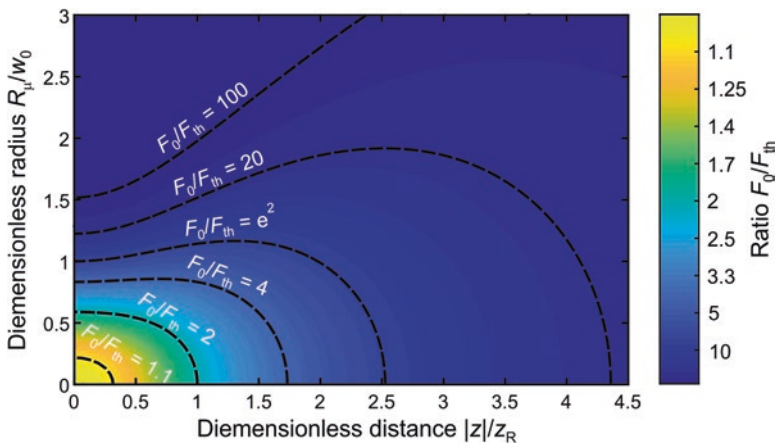


Fig. 11 The theoretical dependence (calculated by Eq. (16)) of the μ -structure radius as a function of the axial distance from the beam's focus for different ratios F_0/F_{th}

$$R_\mu = w_0 \sqrt{\frac{1}{2} \ln \left(\frac{F_0}{F_{th}} \right)}. \quad (17)$$

Equation (17) can be used to calculate the threshold fluence for a given material under given processing parameters. To do this, one should put the surface in the focal plane and should texture the lines or holes at different peak fluences (e.g. by changing the average power at a constant repetition rate). The result for stainless steel is shown in Fig. 110b, where the measured radii are presented by the points. Fitting Eq. (17) across the measured points gives the fluence threshold that in this case equals 3.8 J cm^{-2} . The fit further enables the estimation of the μ -channel diameter at $F_0 = 25.1 \text{ J cm}^{-2}$ that cannot be measured otherwise due to overlapping of the μ -channels. Fit gives that $D_\mu = 56 \text{ }\mu\text{m} > \Delta y$. This calculated result is experimentally confirmed by μ -channel diameter measurements at the same fluence, but for $\Delta x = 100 \text{ }\mu\text{m}$ [134].

Since the surface topography significantly depends on the combination of the peak fluence and scanning line separation, the variation of these two parameters can be used for structuring different surface topographies. In a special case, when D_μ is just slightly smaller than scanning line separation Δy , the μ -cavities appear on the border between two adjacent μ -channels as clearly visible from Fig. 10c, which is the magnification of the selected area (marked by the white rectangle) in the third micrograph of Fig. 10a. As we will show later this approach enables production of the μ -cavities that play an important role in engineering of surfaces for enhanced heat transfer [46, 62].

It should be noted that several definitions of pulse fluence are used in the literature. This may lead to confusion, if the authors do not explicitly state how they calculate (define) the beam fluence. Usually, the fluence is calculated as a pulse energy per area within the beam waist radius

$$F = \frac{E_p}{\pi w_0^2}. \quad (18)$$

However, one should consider that the laser radiation is theoretically not limited to the radius of the beam. This radius is defined as the distance from the centre to the radius, where the beam intensity decreases to the $1/e^2$ peak intensity. Thus only $(e^2 - 1)/e^2 \approx 0.86$ of the pulse energy is included into this area of the beam cross section. The average fluence within the beam radius w thus equals

$$F_w = \frac{e^2 - 1}{e^2} F. \quad (19)$$

The peak fluence F_0 is also commonly provided. In this case the following relation is valid:

$$F_0 = 2F. \quad (20)$$

As we can see by comparison of Eqs. (18), (19), and (20), any type of the fluence definition enables comparison of the results obtained by different authors, but the authors should *always* reveal how they define it.

3.3 The Role of the Scanning Line Separation

Not only the fluence but also the scanning line separation Δy and the scanning strategy determine the surface morphology modifications [104, 128, 134]. Moreover, when one would like to produce surfaces with different roughness, changing the scanning line separation is more appropriate approach as changing the pulse fluence, since pulse fluence – when well above the threshold fluence for laser ablation – has only minor effect on surface roughness, as follows from Eqs. (16 and 17).

The influence of the scanning line separation on surface topography is clearly evident from the SEM micrograph in Fig. 12a that shows the surface morphology of stainless steel for different Δy at a constant fluence $F_0 = 31 \text{ J cm}^{-2}$. It is observable that under very small scanning line separation ($\Delta y < D_\mu$), the processed surface becomes highly porous with no specific (long) μ -channels (Fig. 12a for $\Delta y = 25 \mu\text{m}$). On the contrary, large scanning line separation (i.e. $\Delta y > D_\mu$) leads to the well separated μ -channels, as visible in Fig. 12 for $\Delta y = 200 \mu\text{m}$.

Similar behaviour of the surface morphology as a function of the scanning line separation (at a constant pulse fluence) has been observed by several authors [128, 134, 145], who used the surface roughness parameter S_a as the main evaluation metric of the surface morphology after the laser texturing. They all show (similarly as

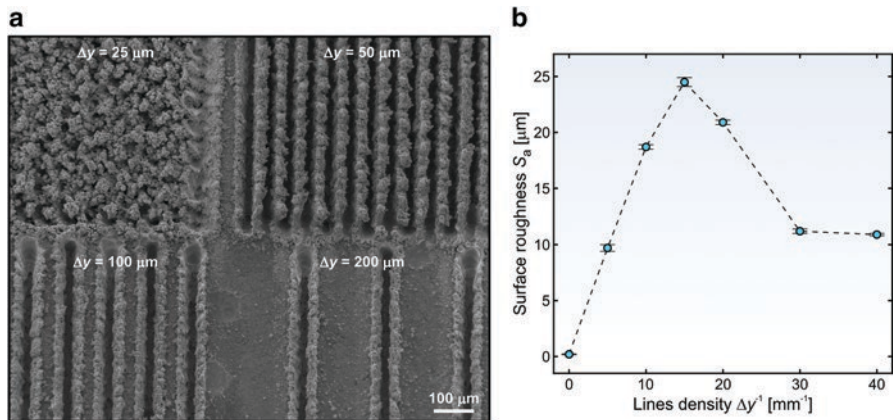


Fig. 12 Influence of the scanning line separation on (a) surface topography and (b) average surface roughness S_a . Note that in (b) the line density Δy^{-1} (i.e. the inverse value of the scanning line separation Δy) is used for better presentation; here, the unprocessed surface corresponds to $\Delta y^{-1} = 0$. (Panel (a): Reproduced and adapted from [104] with permission from Springer. Panel (b): Reproduced and adapted from [128]. Creative Commons Attribution 4.0 International License)

in Fig. 12b) that S_a increases by decreasing Δy until $\Delta y > D_\mu$, since under this condition the laser-textured μ -channels are (well) separated (as for $\Delta y \geq 50 \mu\text{m}$ in Fig. 12a). However, a further decrease of Δy leads to a decrease of the average surface roughness parameter, since S_a measures the arithmetical mean of differences in the height of each point compared to the arithmetical mean of the entire surface. Therefore, it is not sensitive to the increased porosity (as visible for $\Delta y = 25 \mu\text{m}$ in Fig. 12a) induced by a further decrease of Δy [128]. Note that the line density Δy^{-1} (i.e. the inverse value of the scanning line separation Δy) is used for better presentation in the ordinate axis of Fig. 12b. The non-processed surface corresponds to $\Delta y^{-1} = 0$ and in case of Ref. [128], where Fig. 12b is taken from, its surface roughness equalled $S_a = 202.7 \text{ nm} \pm 5 \text{ nm}$.

Higher SEM magnification (the inset in Fig. 13a) reveals that the tops of μ -channels are covered with bumps with the dimensions in the range of 1–7 μm [104]. This bimodal surface is also clearly visible from the cross section in Fig. 13b, while the electron backscatter diffraction (EBSD) analysis of the cross section (Fig. 13c) reveals that both the bulk and the processed material (on the tips) have no specific grain orientation. The austenitic grains of the bulk (unprocessed) material have typical dimensions between 20 and 30 μm , while the laser texturing results in much finer grains with typical dimension below 10 μm [104].

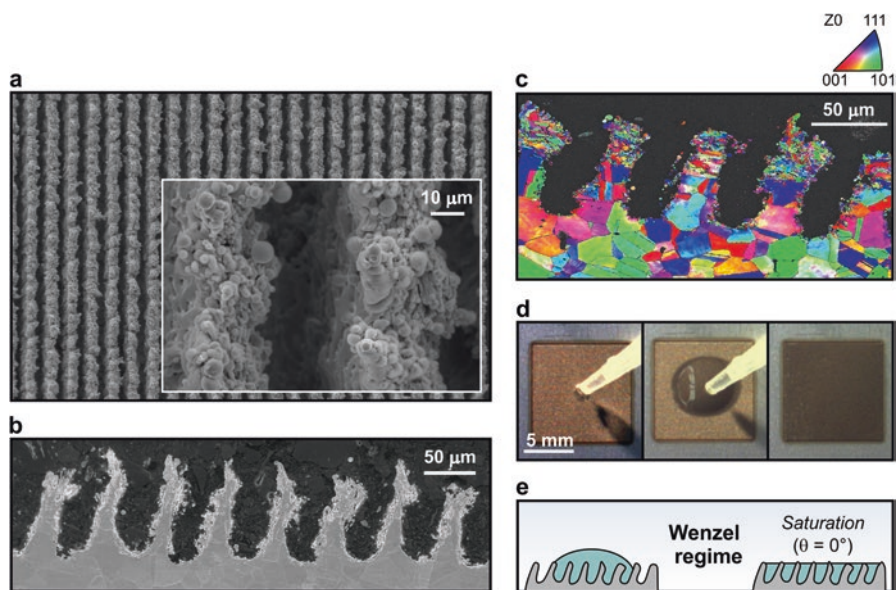


Fig. 13 Stainless steel surface morphology, microstructure and wettability after high fluence laser texturing with $\Delta y = 50 \mu\text{m}$ and $F_0 = 31 \text{ J cm}^{-2}$. (a) SEM micrographs of the surface and (b) its cross section. (c) EBSD measurement (Z direction IPF colouring orientation map). (d) Surface in a saturated Wenzel regime immediately after the laser texturing. (e) Schematic presentation of the saturated Wenzel regime. (Panels (a)–(d): Reproduced and adapted from [104] with permission from Springer)

3.4 Surface Wettability Immediately After Laser Texturing

As shown by several authors [114, 134, 146], the scanning line separation and surface roughness after nanosecond laser texturing significantly influence the surface wettability. Immediately after the laser texturing (with high fluences), the water droplet shows complete wetting of the entire processed surface and forms a thin film, as visible in Fig. 13d. This indicates that laser texturing turns the surface superhydrophilic and causes the appearance of the saturated Wenzel regime with a contact angle $\theta = 0^\circ$ (see schematics in Fig. 13e).

In the Wenzel regime, the liquid-solid interaction results in homogeneous wetting. Thus, the liquid completely penetrates into the solid surface asperities and the Wenzel contact angle θ_w depends on the Young angle θ_Y for an ideal (i.e. flat, perfectly smooth, chemically homogeneous, insoluble, non-reactive, and rigid [48, 147]) surface and the surface roughness r , defined as the area of the rough surface and the area of the corresponding smooth surface [148]:

$$\cos(\theta_w) = r \cos(\theta_Y). \quad (21)$$

The Wenzel state (Eq. (21)) predicts saturation of an ideal hydrophilic surface with the Young angle θ_Y below 90° to a film ($\theta_w = 0^\circ$) when roughness r exceeds the saturated roughness r_s [149, 150]:

$$r_s = \frac{1}{\cos(\theta_Y)}. \quad (22)$$

Gregorčič et al. [104] have measured the Young's contact angle on a polished stainless steel sample before laser processing to be $\theta_Y = 81.6^\circ$. From this value, it follows that the saturated roughness (Eq. (22)) for their material equals $r_s = 6.8$. From the cross section of the surface after the laser texturing (Fig. 13b), they measured that the surface roughness after laser processing equalled only $r = 4.5 < r_s$, yet the surface was already in the saturated Wenzel state. This additionally confirmed that the laser texturing in an oxygen-containing atmosphere changes the surface chemistry and, consequently, the intrinsic contact angle.

They have additionally confirmed this conclusion by the EDS measurements, presented in Fig. 14b. Figure 14a shows a bump with a broken oxide layer – the EDS measurements were performed in points 1–6 and within areas A (without the oxide layer) and B (covered by the oxide layer). The measured weight percentage of the oxygen within the selected points and/or areas is revealed in graph in Fig. 14b. This measurement clearly proves that laser texturing results in surface oxidation.

Formation of oxide layer on metal surfaces significantly affects wetting. Usually, rough metallic oxide surfaces tend to be highly hydrophilic [151]. This explains why the saturated Wenzel regime happens already with roughness smaller than r_s , predicted by Eq. (22) for the Young's angle of the base (unprocessed) material. The laser processing does not only change the surface morphology, but it also

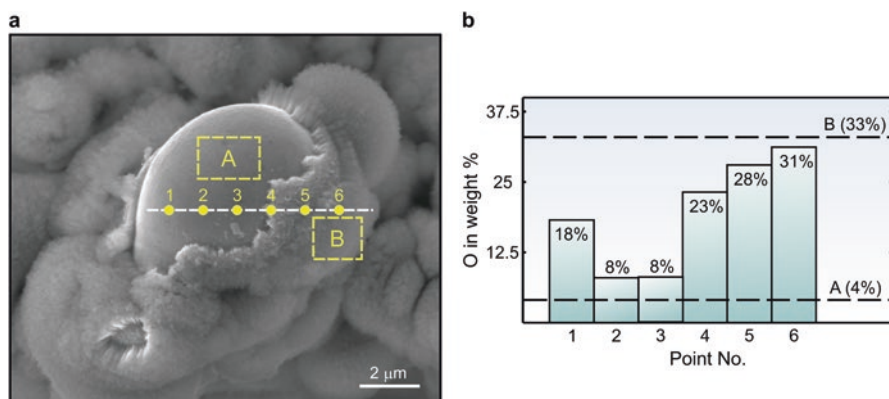


Fig. 14 (a) SEM micrograph of a bump with broken oxide layer on a stainless-steel surface after laser texturing and (b) EDS analysis of the oxygen content element. (Reproduced and adapted from Ref. [104] with permission from Springer)

significantly modifies the surface chemistry [103, 105, 106, 126] leading to a decrease in Young's angle due to metal oxidation. Thus, not only the morphological modification but also the modified surface chemistry increases the metallic surface wettability after laser texturing.

3.5 The (Super)Hydrophilic-to-(Super)Hydrophobic Transition

The initial (super)hydrophilic state of the laser-textured surface is not stable. Several authors [104, 114, 123, 134, 145, 146, 152, 153] have reported that the (super)hydrophilic-to-(super)hydrophobic transition occurs if the laser-textured sample is exposed to atmospheric air for an appropriate period (usually from several days to several weeks). The underlying mechanisms responsible for this transition are still under debate [154]. Different (even contradictory) mechanisms have been proposed, including decomposition of carbon dioxide into carbon with active magnetite [152]; partial surface deoxidation [155] and creation of hydrophobic functional groups [153]; as well as the absorption of organic matter from the atmosphere, where the processed samples are stored (i.e. the hydrocarbon contamination) [151, 156, 157].

We have several reasons to believe that the wettability transition of the laser-textured metallic surfaces happens mainly due to the hydrocarbon contamination, although it seems that contradictory results exist in the literature [105]. Long et al. have shown that the time needed for the (super)hydrophilic-to-(super)hydrophobic transition after laser processing of aluminium [112] and copper [151] remarkably depends on the atmosphere, where the surfaces are stored after the processing. Similar behaviour was observed also by Yan et al. [158] for laser-textured brass that was stored in different media (including liquids) after texturing. They all found that the organic-rich environments significantly shorten the time needed for the

transition toward hydrophobicity. Their XPS analysis also revealed that the C/metal ratio corresponds well with trends in measured contact angles. Larger contact angles always appeared in combination with relatively high C/metal ratio. Additionally, the analysis of the relative atomic percentage of nonpolar C-C(H) bonds that serve as hydrophobic regions suggested that high C/metal ratios and high atomic percentage of C-C/C-H value may be an indicator of a hydrophobic surface [112, 151]. However, it should be pointed out that such wettability transition is not observed solely on the laser-induced oxides, since similar effects due to airborne hydrocarbons have also been discussed by Takeda et al. [159], who studied the increase of the contact angle on metal oxide films deposited on soda-lime-silica glass by magnetron sputtering; by Strohmeier [160], who exposed plasma-treated aluminium foil to the atmospheric air; as well as by Seshardi et al. [161] who studied the effects of gamma (ionizing) irradiation of metallic surfaces on their wettability.

Since the low-temperature annealing could remove the surface modified monolayer [162], different authors annealed at low temperatures the aged laser-textured samples that have already developed (super)hydrophobicity [112, 151, 156]. Their results show that the low-temperature annealing (in the range of 250–350 °C) recovers the initial (super)hydrophilicity. Additionally, the XPS measurements [112, 156] after the annealing process revealed a *decrease* of C/metal ratios as well as *decreased* content of C-C/C-H bonds. This leads to a conclusion that thermal annealing partially removes the adsorbed organic contaminants.

Several authors have convincingly demonstrated that spontaneous (super) hydrophilic-to-(super)hydrophobic transition under ambient conditions happens for a number of laser-textured metals and metal alloys with different chemical and catalytic activity as well as different crystallographic structure. These samples were textured by femtosecond [152, 163], picosecond [112] and nanosecond [104, 114, 134, 146] laser pulses. Moreover, similar transitions have also been observed on oxides that were not induced by laser radiation [159–161]. All of this indicates that the mechanism of wettability transition after laser texturing should be universal and should not characteristically depend on a specific metal or metal alloy as well as on specific (laser-)processing parameters [154].

However, some observations reported in the literature are at the first sight not consistent with the above-reviewed results. In this context, Chun et al. [164], Ngo et al. [165] and Lian et al. [166] reported that they *accelerated* the hydrophilic-to-hydrophobic transition of laser-textured samples by low-temperature annealing. Unfortunately, they do not reveal enough details of the chemical analysis (of a thin surface layer), which would explain, why they observed the exact opposite of what was shown very convincingly by Long et al. [112, 151]. Thus, we have repeated similar low-temperature annealing experiment with (i) fresh, still superhydrophilic laser-textured surfaces and with (ii) aged, already superhydrophobic laser-textured surfaces [105]. If the furnace was *free of contaminants*, the result of the low-temperature annealing was *always* a hydrophilic surface. When a fresh, still hydrophilic surface was annealed, the water repellency has not developed, while the low-temperature annealing led to increased wettability (turned off the hydrophobicity) on aged, already hydrophobic laser-textured surfaces. On the contrary, if the

furnace was *contaminated* by organic compounds, the low-temperature annealing universally (i.e. independently on metal used) resulted in hydrophobic surface [105].

Figure 15a shows the stainless steel surface, processed by the parameters described in Ref. [104], immediately after the texturing, when the surface was in a saturated Wenzel state ($\theta = 0^\circ$). The SEM image of the same surface in a superhydrophobic state ($\theta = 153^\circ$, the roll-off angle $< 7^\circ$) 3 months later is shown in Fig. 15b. The comparison of these two images reveals that the suggestion of some authors [114] that surface morphology does not change during aging the samples might only be true on the micrometre scale and not necessarily on a sub-micrometre scale. Figure 15 clearly demonstrates the development of an additional nanostructure on the oxide layer in the scale of 200–700 nm. In this example, the nanostructure immediately after the laser-texturing looks like a “closed flower”, while 3 months later, it looks like an “opened flower”. Although this sample was already superhydrophobic 30 days after the laser texturing, the nanostructure was still developing in a fractal-like manner, as shown on the SEM image acquired on the same sample 10 months later (Fig. 15c).

To additionally confirm the results of low-temperature annealing experiments by Long et al. [112, 151], this superhydrophobic sample was put into an uncontaminated furnace at 350°C for four hours. After the low-temperature annealing, the superhydrophobicity was turned off, and the surface was returned into the saturated Wenzel regime ($\theta = 0^\circ$). The morphology on the micrometre and nanometre scale has not changed significantly, as visible from Fig. 15d, while the content of C atoms was significantly reduced. This suggests that the mechanisms proposed by Long et al. for aluminium [112] and copper [151] surfaces are also valid for stainless steel. Moreover, it additionally confirms that the wettability transition after laser texturing is governed mainly by the surface chemistry modification. The (sub-micrometre) morphology modification during surface aging is not negligible, but it seems that its influence on the overall wettability behaviour is insignificant.

Similarly, at the first sight contradictory effect is observed, when the laser-textured surfaces are exposed to vacuum [151, 167]. Long et al. [151] reported that

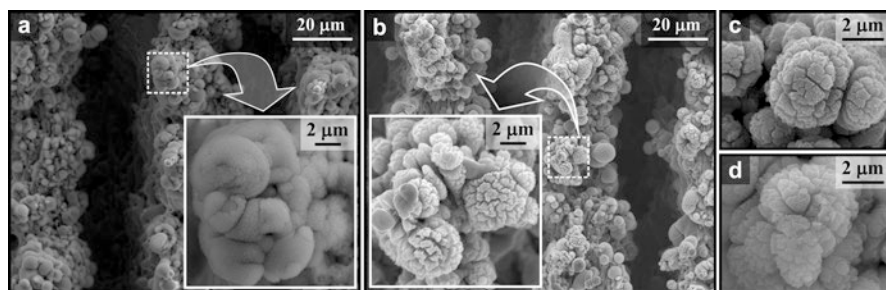


Fig. 15 SEM micrographs revealing the surface nanostructure evolution of stainless steel: (a) one day after laser texturing ($\theta = 0^\circ$); (b) 3 months after laser texturing ($\theta = 153^\circ$); (c) 10 months after laser texturing and before low-temperature annealing ($\theta > 150^\circ$); (d) sample from (c) after low-temperature annealing ($\theta = 0^\circ$)

“vacuum” accelerates the hydrophilic-to-hydrophobic transition just like an organic-rich atmosphere. This corresponds well with findings by Yan et al. [168], who examined the wettability of a Fe_2O_3 nanoflakes film. They observed a remarkable acceleration of the temporal contact angle increase, when the film was exposed to vacuum. In this case, the surface became ultrahydrophobic with a contact angle of 160° , compared to 0° for the sample that was stored in a nitrogen atmosphere. They explained that this happens due to the chemisorption of organic molecules such as oil droplets from vacuum pumps.

These findings clearly show that the contamination of the atmosphere, where the surfaces are aged and/or annealed, plays a significant role in the wettability transition. Moreover, the existing experimental results also clearly indicate that the *ambient conditions* should be appropriately controlled during aging of the samples and that an analysis of surface chemistry is required to correctly interpret the experimental observations.

As shown by Gregorčič et al. [134], the *long-term examination* of wettability transition after laser texturing is another important factor that should be taken into account to develop pertinent conclusions. In this study, the authors compared the contact angles after a short- (up to 40 days), intermediate- (100 days) and long-term (1 year) superhydrophilic-to-(super)hydrophobic transition of laser-textured stainless-steel surfaces exposed to atmospheric air. The measured contact angles as a function of time for different scanning line separations are shown in Fig. 16. According to Eq. (12), smaller scanning line separation means higher total energy irradiated to a surface. The results in Fig. 16 clearly indicate that solely the short-term consideration of the wettability transition (many authors [104, 114, 123, 133, 146, 153, 169] that usually indicate “stable conditions” after 1 or 2 months of the contact angle analysis) may lead to the wrong conclusion that the “final” contact angle is strongly influenced by the scanning line separation, since in the short-term period, the smallest Δy (Fig. 16a) results in a still hydrophilic surface. Such a conclusion was developed by Ta et al. [146] who evaluated the surface wettability for only 15 days following laser texturing. However, as shown by Gregorčič et al. [134], an intermediate-term evaluation (i.e. after 3 months) already reveals that such hydrophilic state is not stable, since hydrophobicity is achieved on all the samples, independent of the scanning line separation (see the contact angles within “intermediate-term” in Fig. 16). Moreover, a long-term examination of the wettability transition reveals that after one year, all the samples expressed a successful transition from superhydrophilic to superhydrophobic state. After the long-term period, even the surfaces that were processed by the smallest scanning line separation (Fig. 16a) achieved a lotus leaf-like state with $\theta > 150^\circ$ and a roll-off angle $< 5^\circ$ [134].

In contrast to the short-term evaluation [146], the long-term results in Fig. 16 suggest that the highest final contact angle is achieved for the highest value of the total absorbed energy per unit area, defined by Eq. (12). A long-term evaluation of the wettability transition is especially important, when high total energy per unit area is used due to (i) high average power (P in Eq. (12)), (ii) slow processing speeds (v in Eq. (12)), and/or (iii) small scanning line separations (Δy in Eq. (12)). In this case, immediately after laser texturing, the surface is in a highly saturated

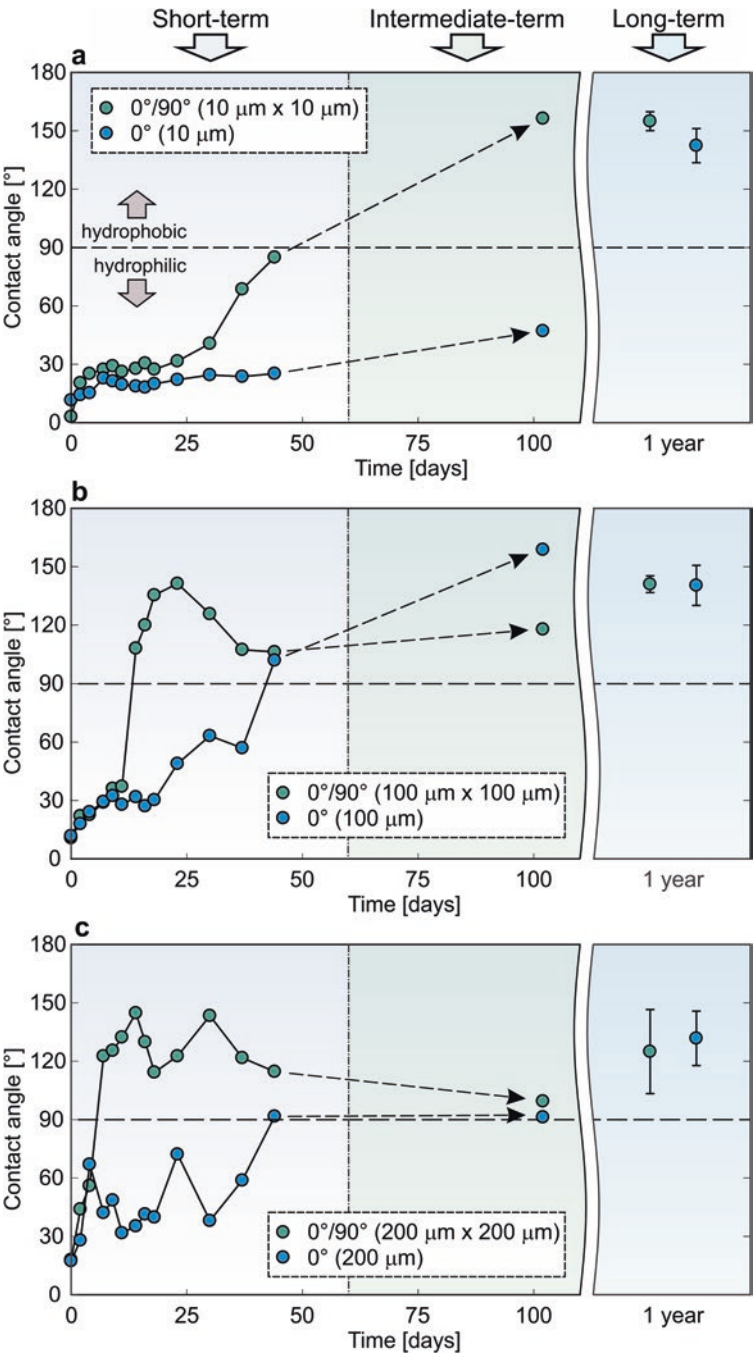


Fig. 16 Short-term, intermediate-term and long-term static water contact angle development on surfaces with the following scanning line separations: (a) $\Delta x = \Delta y = 10 \mu\text{m}$; (b) $100 \mu\text{m}$; and (c) and $200 \mu\text{m}$. The results are shown for the 0° texturing (blue points) and for the $0^\circ/90^\circ$ texturing (green points) with $F_0 = 25.1 \text{ J cm}^{-2}$. (Reproduced and adapted from Ref. [134]. Creative Commons Attribution 4.0 International License)

Wenzel state ($\theta = 0^\circ$), making it impossible to evaluate different surface wettabilities through the contact angle. Thus, other approaches for the wettability characterization (e.g. measuring the wicked volume flux [44] or droplet spreading [170]) should be used to distinguish different wettabilities of such surfaces immediately after the texturing. As shown by the results in Fig. 16, a higher amount of total absorbed energy per unit area does not lead only to a more hydrophilic surface, but it also causes a *slower* development of hydrophobicity. However, the slower development usually results in a *higher* final contact angle (i.e. a more hydrophobic surface) [134].

3.6 Fabrication of Multiscale μ -Cavities by Laser Texturing

- As we have discussed in the Sect. 2, the surface wettability significantly influences the boiling performance. However, there are several reasons why solely the modification of surface wettability cannot represent the ultimate heat transfer enhancement approach in the nucleate boiling applications.
- The wettability significantly depends on fluid properties, and different working fluids are used in a diversity of boiling applications. Water, a polar liquid with high surface tension, is used in boiling water nuclear reactors, while dielectric, non-polar liquids with almost one order of magnitude lower surface tension and small capillary lengths are usually employed for phase-change heat transfer in heat pipes and refrigeration systems.
- As has been shown by Može et al. [49], the surface wettability may change during boiling due to intense processes, such as the vaporization of the working fluid, high surface temperatures and high frequency temperature variations due to bubble nucleation (further discussed in Sect. 4.5).
- As already described, the wettability can also change due to hydrocarbon contamination, which does not happen only in contaminated gas atmospheres [112, 151] but also in liquid atmospheres [158].
- When surfaces are exposed to ionizing radiation, this also significantly influences surface wettability [161, 171, 172].

Therefore, when developing different functionalization approaches for surfaces in boiling heat transfer applications, it is highly desirable that such functionalization will also decrease the influence of the wettability on boiling performance and stability. In this context, Zupančič et al. [62] have shown that surfaces with laser-induced multiscale μ -cavities enable enhanced and controlled saturated nucleate boiling performance. This was proven further by Gregorčič et al. [46], who successfully used such surfaces for enhanced and controlled boiling heat transfer in a wide range of fluids (further discussed in Sect. 4.3), independent of their polarity or surface tension, as well as by the experiments by Zakšek et al. [84], who demonstrated that this kind of surfaces enable controlled boiling also in binary liquid mixtures. All these results confirm that functionalization of the heating surfaces by multiscale μ -cavities

can decrease the influence of the surface wettability on the key boiling heat transfer parameters and, consequently, leads to more stable heat transfer processes that are independent of (i) fluid properties, (ii) hydrocarbon contaminants and (iii) ionizing radiation.

Two possible strategies can be utilized to produce surfaces with multiscale μ -cavities by (fibre) laser texturing in a straightforward, compact, robust, flexible, chemical-free and cost-effective way. The first one is *the very high fluence approach*. When high pulse fluences are used, laser ablation leads to a high amount of remolten and solidified material around the μ -channels, as visible in Fig. 17a. Here, the μ -cavities with *diverse* radii appear due to significant ejection of this remolten material. However, this strategy is not appropriate for processing thin foils (e.g. with a thickness of 25 μm) that are required when measuring the transient temperature fields using an IR camera on the backside of the foil, as performed in several experimental studies [34, 46, 62, 84, 174], since very high fluences may lead to up to 100 μm deep μ -channels (Fig. 13b). Even so, this strategy can be successfully employed in experiments with heating blocks [49, 175] as well as in real production including modification of different types of heat exchangers, where much thicker surfaces are used.

The second approach is *the (variable) scanning lines approach*. Here an appropriate combination of (lower) pulse fluences and scanning line separation, as suggested in Fig. 10c, is used. Typical surface microstructures obtained by this approach are shown in Figs. 18b–e. However, using this strategy, only small variations in scanning line separation and/or pulse fluence can lead (according to Eq. (17) in combination with the equidistant spacing sketched in Fig. 9a) to (i) closed gap

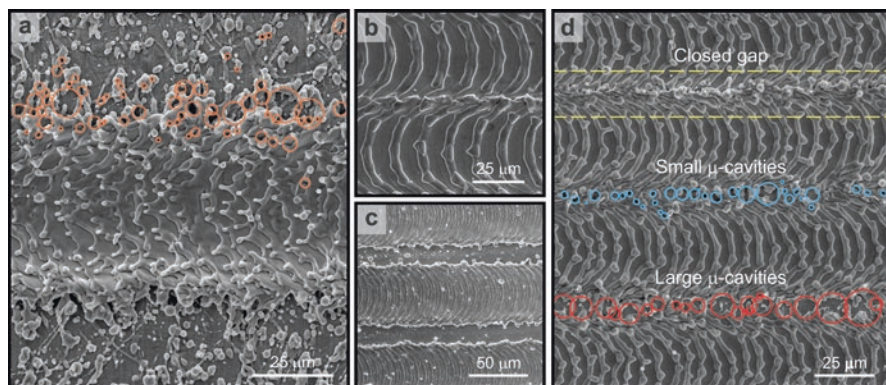


Fig. 17 (a) Surface modification using the very high fluence texturing approach. The identified μ -cavities on the top edge of the μ -channel are marked by the orange circles with their radii corresponding to the largest dimension of the identified μ -cavity. The variable scanning approach with (b) closed gap without μ -cavities, (c) gap that is too wide and thus lacks appropriate μ -cavities and (d) with multiple scanning line separations leading to higher diversity of the μ -cavities' radii. (Reproduced and adapted from Ref. [173]. Creative Commons Attribution 4.0 International License)

without μ -cavities due to too small distance between two consecutive μ -channels (Fig. 17b) and (ii) too wide gap lacking appropriate μ -cavities (Fig. 17c).

Although lower fluences may be used with the *scanning lines approach*, they should not be too low, since this leads to less ejection of the remolten material. In this case, the μ -channel edge is smooth and even overlap will not lead to appropriate μ -cavities. An example of this situation is demonstrated in Fig. 17c, where very smooth edges of the μ -channels are clearly visible. The surfaces from Fig. 17c, d were processed with the same laser parameters, but the beam diameter at the focusing lens was changed. In case of the surface on Fig. 17c, the beam diameter on the F-theta lens D equalled 5 mm, while in case of Fig. 17d, it equalled 7.5 mm. According to Eq. (8), the ratio between the beam waist radii, therefore equalled 1.5, which means that (according to Eq. (18)) the surface on Fig. 17c was processed with 2.25 times lower fluence as the surface in Fig. 17d.

- The robustness of the (variable) scanning lines approach can be significantly improved by using multiple scanning line separations (e.g. see Fig. 9b), as

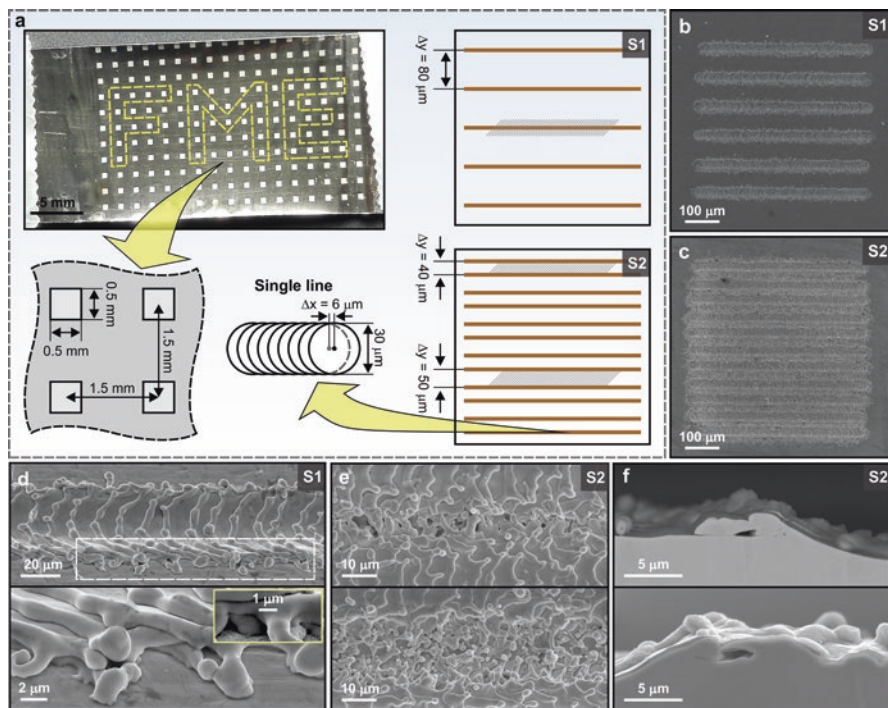


Fig. 18 (a) Stainless steel foil with laser textured array and schematic presentation of the laser-texturing method. SEM images of the square, textured by (b) S1 and (c) S2 structure. Magnified SEM images of (d) S1 structure [marked area in S1 in (a)] and (e) S2 structure on the border between two channels [marked areas in S2 in (a) – top for $\Delta y = 40 \mu\text{m}$, bottom for $\Delta y = 50 \mu\text{m}$]. (f) Cross section of S2. (Reproduced from Ref. [46]. Creative Commons Attribution 4.0 International License)

reported by Gregorčič et al. [46], where the surface was processed by two different Δy . This strategy makes it possible also to use low-cost scanners with lower angular (positioning) resolution. Moreover, such approach additionally leads to a higher diversity of μ -cavities' radii and, thus, enables higher degree of enhancement of the boiling process (according to the theoretical results presented in Fig. 3).

- To demonstrate the boiling performance of such surfaces, Gregorčič et al. [46] fabricated two types of structures on stainless steel foils. The first one was labelled S1 and consisted of well-separated channels, as shown in Fig. 18b. The other one was labelled S2 and was fabricated using a scanning line separation that alternated between two values, as shown in Fig. 18c. A SEM image, acquired on the surface S1, tilted for 60° , reveals that μ -cavities are formed even on the edges during the solidification of the remolten material after laser ablation (Fig. 18d, the white dashed rectangle). This indicates that the very high fluence approach is additionally effective, since the μ -cavities do not appear only on the top of the surface but also at the intersection between the drilled μ -channel and the ejected, solidified material. As was shown in [46], the radii of μ -cavities, induced on well-separated μ -channels with lower fluences, are typically in range from approximately hundred nanometres up to $1\text{ }\mu\text{m}$. The situation is slightly different for S2, where the scanning line separation is decreased to the values just slightly larger than the channel width. Here, the range of μ -cavity radii is significantly increased due to overlapping of the solidified material at the edges of the neighbouring channels (Fig. 18e). Thus, the radii go from approximately hundred nanometres up to $5\text{ }\mu\text{m}$. Additionally, this approach results in a more porous structure as the cavities are much deeper compared to the well-separated μ -channels, where the ejections of the solidified material from parallel μ -channels do no overlap. The overlapping of the solidified material may also lead to interconnected μ -cavities, as revealed by a surface cross section in Fig. 18f.

4 Applications of Laser Engineered Surfaces in Pool Boiling

4.1 Experimental Boiling Performance Determination

Evaluation of nucleate pool boiling performance on different surfaces requires a carefully designed experimental setup that allows accurate monitoring of the boiling surface temperature (wall temperature) and the heat flux while maintaining the homogeneity of the bulk liquid at the desired pressure and temperature. In the most common experimental configuration, the boiling surface is heated through conduction from an external heating source, and the heat flux and the wall temperature are calculated from several temperature measurements along the heated block [40, 49, 175–178]. This configuration is suitable to determine the spatio-temporal averaged

wall temperature and heat flux in order to obtain reliable values of the HTC and CHF that are also observed in real applications.

However, to study specific parameters like nucleation frequency, distribution of the active nucleation sites, bubble growth characteristics and local heat fluxes, the transient wall temperatures on the boiling surface should be determined. This can be achieved using the liquid crystals [179, 180], microthermocouple arrays [181, 182], temperature sensitive paints [183, 184], fluorescence microscopy [69] or infrared (IR) thermometry [30, 70–72, 185, 186]. Since the IR thermometry is used most commonly, it is a well-developed technology able to detect small temperature differences (~ 20 mK) with pixel-to-pixel resolution down to about $10\text{ }\mu\text{m}$. To successfully detect transient temperature fields underneath a growing bubble, the heater should have relatively small thermal capacitance, which is generally achieved by depositing a thin metal layer atop of an IR transparent substrate or by using a thin metal foil. Boiling on Joule-heated metal foils supported by the high-speed IR thermography (framerate ≥ 1 kHz), which is specifically studied in this section (Fig. 19), can provide an accurate estimation of the wall temperature, bubble footprint area and its growth time, locations of active nucleation sites and local heat fluxes [187]. However, the measured CHF values on such foils (usually their thickness is below $50\text{ }\mu\text{m}$) are significantly smaller compared to thicker heaters due to conductance/capacitance effects [188, 189]. After optimizing a specific surface structure on a thin heater, the actual CHF value should be experimentally determined on the heater with the thickness above the asymptotic limit (with thickness typically above $500\text{ }\mu\text{m}$).

4.2 Laser-Induced μ -Cavities as Active Nucleation Sites

Based on the nucleation criteria explained in Sect. 2.1, the optimal cavity radius for water on superhydrophilic surfaces is within the range of $1\text{--}10\text{ }\mu\text{m}$. This is below the generally achievable spatial resolution of the IR thermography, which is limited by diffraction. Despite that, Zupančič et al. [62] demonstrated that appropriate post-processing of IR data can be used to confirm the effectiveness of μ -cavities. They laser-textured a stainless-steel surface (Fig. 20a) by using the $0^\circ/90^\circ$ texturing with a variable spacing (Fig. 9d), where Δy varied from 10 to $200\text{ }\mu\text{m}$ in both directions (the left-hand side of Fig. 20a). In the regions, where the scanning line separation was in the range of $30\text{--}50\text{ }\mu\text{m}$, the multiscale μ -cavities were induced. Their existence was confirmed by SEM micrographs, and their radii were measured to be in the range of $0.1\text{--}3\text{ }\mu\text{m}$ (SEM images in Fig. 20a), while the surface exhibited a non-uniform wettability with contact angles ranging from 66° to 142° .

Contrary to (super)hydrophilic surfaces, superhydrophobic ones would theoretically require cavities with radii in the range of $100\text{--}1000\text{ }\mu\text{m}$. However, low thickness of the foil prevents the fabrication of such large cavities with appropriate depth. Therefore, only cavities in the micrometre range are significant in the described situation. Post-processing algorithm of the transient temperature fields, acquired by

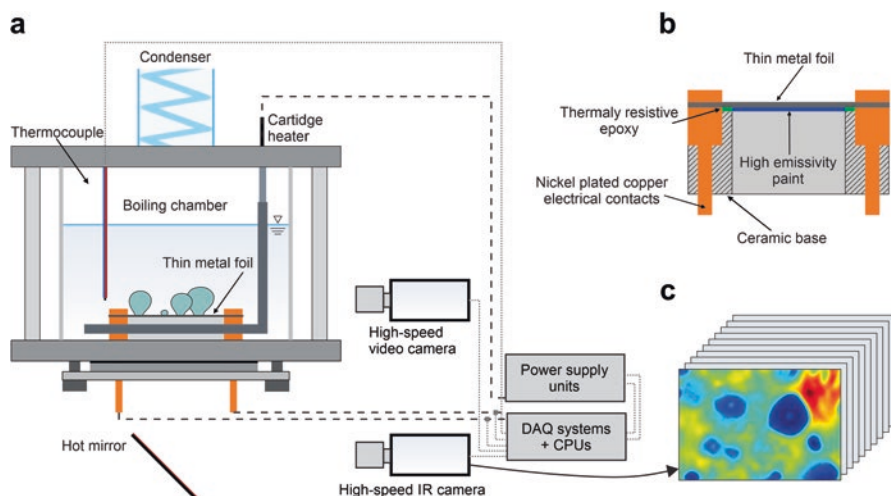


Fig. 19 (a) A simplified schematics of the pool boiling experimental setup for thin metal heaters and (b) cross section view of the thin-metal-foil heater unit. (c) Representation of consecutive temperature fields of the boiling surface obtained by the high-speed IR camera – note that cooler regions are represented by blue colour and therefore indicate the bubble footprints

the IR thermography, is explained in Fig. 20b. The result of the processing in Fig. 20c clearly shows that the active nucleation site density is periodically distributed across the entire surface and exactly matches the spatial distribution of the μ -cavity regions. The presence of the μ -cavities increases the active nucleation site density, lowers the onset of nucleate boiling for more than 5 K compared to bare stainless steel and improves the overall HTC for more than 110%. The combined effect of μ -cavities, increased surface roughness and modified wettability increased the nucleation frequency for at least 200% and nucleation site density for two orders of magnitude compared to untreated material [62]. Slightly lower enhancement was also observed by Kruse et al. [14], who studied nucleate pool boiling on stainless steel surfaces (type AISI 304) processed by femtosecond laser pulses. The superhydrophilic sample S4 (Fig. 20d) incorporated circular pits (cavities) with radii up to about 5 μm .

4.3 *Decreased Influence of Surface Wettability on μ -Cavity-Induced Boiling Performance Enhancement*

The ability of the surfaces with multiscale μ -cavities to provide control over the boiling process in a diverse range of fluids, independent of their polarity and surface tension, was demonstrated by Gregorčič et al. [46]. They fabricated two surfaces (labelled S1 and S2) using nanosecond laser pulses and different scanning line separations, as shown in Fig. 18. The first surface (S1) was textured with well-separated

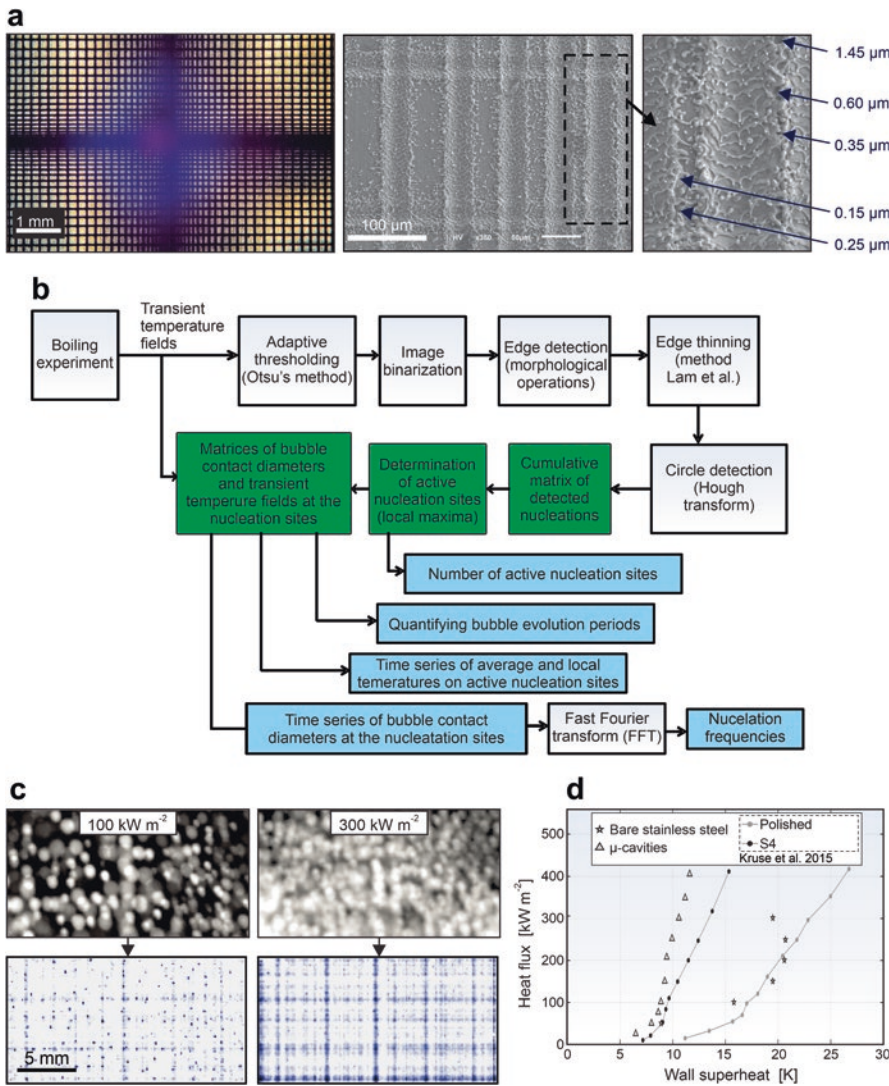


Fig. 20 (a) Microscope image of laser-textured, 25- μm -thick stainless-steel foil with variable scanning line separation and SEM micrograph of the μ -cavity regions with some representative values of cavity radii. (b) Flowchart of the data processing algorithm to detect the active nucleation sites. (c) Cumulative matrix of detected bubble footprints (top) and distribution of active nucleation sites (bottom) over the entire laser-textured foil. (d) Boiling curves during the saturated pool boiling of water for bare stainless steel and laser-textured stainless-steel foil measured up to 400 kW m^{-2} . Results using femtosecond laser-textured surfaces reported by Kruse et al. [14] are shown for comparison. (Panels (a), (c) and (d): Reproduced and adapted from Ref. [62] with permission from Elsevier)

μ -channels, thus containing only small μ -cavities with radii in the range of 0.1–0.75 μm . On the contrary, the variable scanning line separation used for S2 resulted in multiscale μ -cavities with radii in the range 0.1–5 μm . To demonstrate the capability of such surfaces to control boiling, a stainless steel foil (type AISI 316 L) was textured in regions of $0.5 \times 0.5 \text{ mm}^2$ squares forming the shape of the “FME” (Faculty of Mechanical Engineering; see Figs. 18a and the top of Fig. 21).

The textured surfaces were tested in two very contrasting fluids: water, which is polar with high surface tension (58.9 mN m^{-1} at T_{sat} and 1 atm) and high heat of vaporization, and the non-polar, dielectric tetradecafluorohexane (FC-72) with low surface tension (8.0 mN m^{-1} at T_{sat} and 1 atm) and much lower heat of vaporization. It should be emphasized that FC-72 exhibits extremely low contact angles with most practical materials (also superhydrophobic materials) except of some special superrepellent surfaces with re-entrant structures [190]. Although the boiling performance of FC-72 is completely different compared to water, experiments in Ref. [46] convincingly show that surface structure with a wide range of μ -cavity size can provide control over the nucleation locations for different types of fluids with completely different wetting characteristics. The time-averaged temperature fields of the boiling surface in Fig. 21 show the comparison of the active nucleation sites distributions on both surfaces for water and FC-72.

The S1 structures with smaller cavity radii in the range of 0.1–0.75 μm was only able to provide the active nucleations for FC-72 (the left-hand side in Fig. 21), since this range of the μ -cavities radii is not optimal for water according to the Hsu’s nucleation criterion (Eq. (3)), but falls within the desired range for FC-72 and other low-surface-tension fluids. On the other hand, the S2 structure with one order of magnitude larger range of the μ -cavities radii (from 0.1 to 5 μm) resulted in clearly

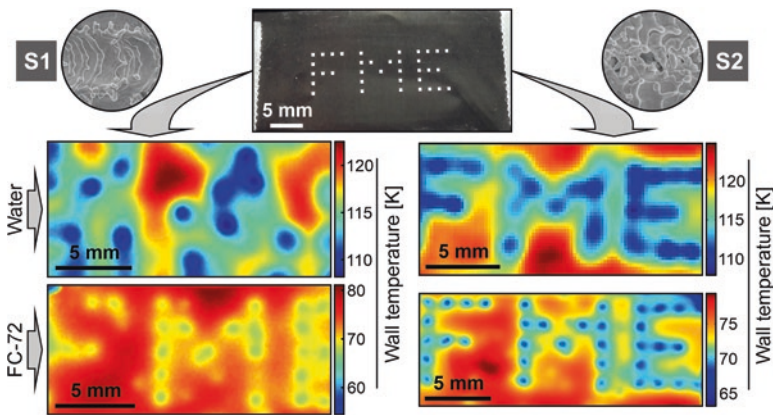


Fig. 21 Controlled nucleate boiling performance of water and FC-72 on laser textured “FME” pattern. Time-averaged temperature fields were taken at 150 kW m^{-2} for water and 15 kW m^{-2} for FC-72. On the laser-textured regions, the static contact angle of water equalled around 16° and for FC-72 around 13° . The S2 structure provides on average 11 K (for water at 150 kW m^{-2}) and 7 K (for FC-72 at 15 kW m^{-2}) lower surface temperature compared to the surrounding inactive regions. (Reproduced from Ref. [46]. Creative Commons Attribution 4.0 International License)

visible “FME” structure for both fluids as a low-temperature region due to constant boiling activity. In addition to water and FC-72, similar behaviour was also confirmed with pure ethanol and water-ethanol mixtures [83, 84].

In addition to stainless steel [14, 46, 62, 191], laser texturing for enhanced boiling applications can be also applied to other important materials, like copper [49, 192], silicon [193], Inconel [192], aluminium, titanium and zirconium alloy. Figure 22a shows an example of the laser-textured zirconium alloy type 702. The textured areas with dimensions of $0.5 \times 0.5 \text{ mm}^2$ are distributed in a hexagonal pattern to reach an optimal filling factor. Similar to the previous example, each textured area consists of multiscale (interconnected) μ -cavities with radii ranging from 60 nm up to 3 μm . These areas were superhydrophilic prior to boiling experiments due to the reasons explained in Sect. 3.4. However, superhydrophilic surface is not ideal to lower the temperature of the onset of nucleate boiling. Despite this, the boiling initially took place only on the μ -cavity regions as presented in Fig. 22b. Significant enhancement of the nucleation site density, nucleation frequency and equal distribution of the nucleation sites on Zr- μC sample resulted in 3–5 K lower superheat (Fig. 22c) and significantly smaller wall-temperature deviation (Fig. 22d) in comparison to the untreated surface. Light-tailed and narrow probability density function is a clear indicator of stable boiling performance [194], which is required to maintain relatively homogenous surface temperature. This is especially important for applications, such as cooling of microelectronic chips.

Figures 21 and 22 convincingly demonstrate that laser surface engineering can be used to develop surfaces with well-defined nucleation sites on diverse materials and for a wide variety of fluids. This represents a tremendous capability to study the optimal distribution of active nucleation sites in the context of (i) achieving a uniform temperature field and high heat transfer rates at low superheats and (ii) delaying the horizontal coalescence of bubbles that prevents a generation of large vapor patches (i.e. to reach high CHF). Some studies [97, 174, 195] have shown that the optimal distance between the active nucleation sites (regions) that leads to the maximal enhancement of the HTC and CHF roughly corresponds to the capillary length of the working fluid. However, a possible deviations from this theory [84] might be attributed to the fact that the growth time, nucleation frequency and bubble size are not the same for all nucleation sites. Recent work by Zhang et al. [196] considered this near-wall stochastic behaviour of individual bubbles and proves that the boiling crisis is a percolative and scale-free phenomenon that can be delayed by optimization of the nucleation site density, the product of the nucleation frequency and bubble growth time ($t_g f_b$) and the time-dependent bubble footprint radii without additional consideration of macroscale hydrodynamic instabilities or microscale fluid-solid interactions. This (rather radical) idea could completely change the future approach in finding the surface engineering solution(s) to optimally enhance the boiling performance. In this context, the demonstrated capability of laser texturing to create well-defined nucleation sites is worth to be investigated further.

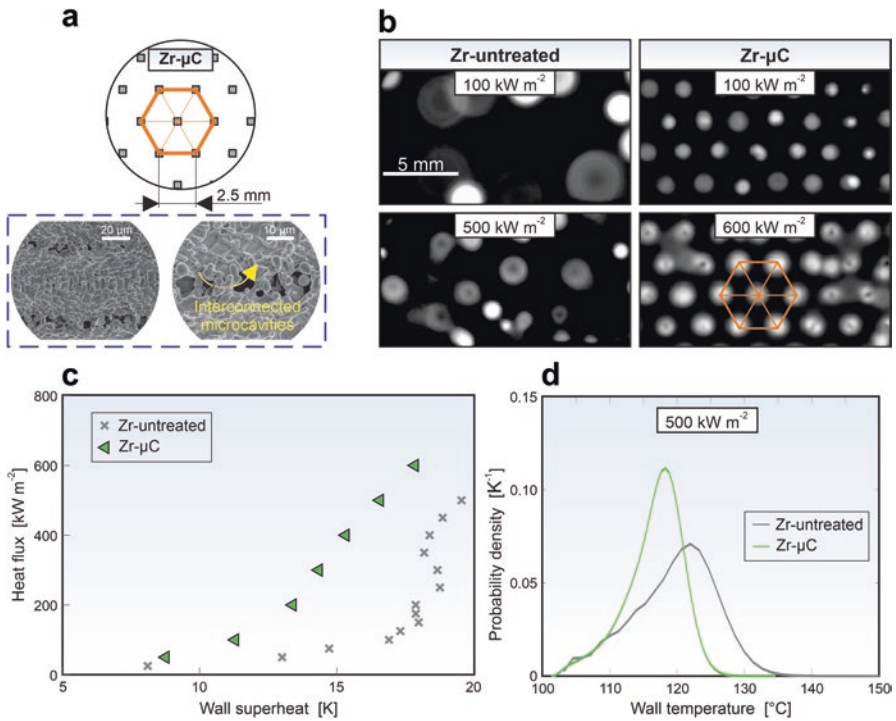


Fig. 22 (a) Laser-textured pattern design on zirconium alloy and SEM images of corresponding μ -cavities. (b) Cumulative matrix of detected bubble footprints on untreated and laser-textured samples at two different heat fluxes. (c) Boiling curves during saturated pool boiling up to 600 kW m^{-2} (note that untreated samples experienced burnout at roughly 540 kW m^{-2}) and (d) wall-temperature distributions for both samples at 500 kW m^{-2}

4.4 Pool Boiling on Laser-Textured Superhydrophobic Surfaces

Numerous publications have shown [34, 79, 81, 197, 198] that superhydrophobic surfaces significantly reduce the temperature at the onset of boiling and the CHF, which was often observed immediately after the boiling incipience. For that reason, superhydrophobic surfaces were not considered to be a good choice in practical (high heat flux) applications. Since the initially superhydrophilic laser-textured surfaces tend to become superhydrophobic over time due to the reasons discussed in Sects. 3.5 and 3.6, the boiling behaviour on surfaces with time-dependent wettability has to be carefully analysed. To evaluate this behaviour, the laser texturing was initially performed on three 25- μm -thick stainless steel foils (type AISI 316 L) to create μ -cavities over the entire surface. The surfaces were then aged at room temperature or in a *contaminated* furnace at 100°C .

Sample, labelled as SHPO, was left exposed to the normal open atmosphere for 82 days and became superhydrophobic with a static contact angle of 158° (Fig. 23b). A similar contact angle of 154° was achieved on the sample SHPO*, which was kept in a contaminated furnace for 17 h, while the sample HPI reached a contact angle of 34° after 2.5 h in a contaminated furnace. As discussed in Sect. 3.5, the aging of the laser-textured metallic surface results in a formation of a thin film of hydrophobic organic compounds due to airborne hydrocarbon, while during the low-temperature annealing, the contamination in form of a thin polydimethylsiloxane (PDMS) coating arose from the silicone components of the furnace [105].

Figure 23a shows the results of two consecutive pool boiling experiments for each sample, first up to 500 kW m^{-2} and second up to the burnout point. Both superhydrophobic surfaces exhibited the same boiling behaviour. During the first run (open circles in Fig. 23a), the onset of boiling was reached at only about 3 K of superheat ($< 25 \text{ kW m}^{-2}$) and provided significant HTC enhancement compared to the untreated sample. However, in the second run (full circles in Fig. 23a), the onset of boiling was increased to about 10 K of superheat, and both boiling curves follow a slope otherwise unexpected for superhydrophobic surfaces until they reached the burnout at $\sim 1300 \text{ kW m}^{-2}$. On the contrary, the HPI sample exhibited the same trends during both experimental runs and provided lower HTC and CHF compared to the superhydrophobic samples.

Such behaviour can be a consequence of several possible reasons. First of all, it was experimentally determined that the contact angle after the first and the second boiling run decreases to about 25° on all laser-textured surfaces. This demonstrates that the naturally occurring superhydrophobicity on the macroscopic scale is not stable during boiling applications. Nevertheless, both superhydrophobic samples provided overall enhancement in HTC in comparison to the hydrophilic one. This indicates that some (super)hydrophobic micro-/nanostructures may be stable and still present on the surface. This kind of structures could lower the nucleation temperature, but their existence cannot be confirmed solely by the macroscale contact angle measurements. Thus, further surface analysis on different scales, including the atomic and molecular scale, is required.

The second important observation is the difference in the onset of boiling temperature between the first and the second experimental run for superhydrophobic samples. Figure 23c,d shows that the early nucleate boiling during the first run takes place at multiple nucleation sites, while the bubbles nucleate at relatively high frequencies and cause small surface temperature fluctuations ($\pm 1 \text{ K}$). However, during the second run, the nucleation site density is decreased, bubble activation temperatures increase by about 5 K, and the temperature profile underneath the bubble follows the recognisable pattern as previously shown in Fig. 4. This behaviour may be explained by the influence of entrapped gasses on the surface before it is exposed to the boiling process for several hours. Shen et al. [199] proved that presence of the non-condensable gas on the hydrophobic surface causes an early transition to nucleate boiling and results in completely different bubble dynamics compared to a fully degassed surface. Additionally, Allred et al. [60] explained that initial Cassie-Baxter wetting state on a superhydrophobic surface results in low surface superheat and

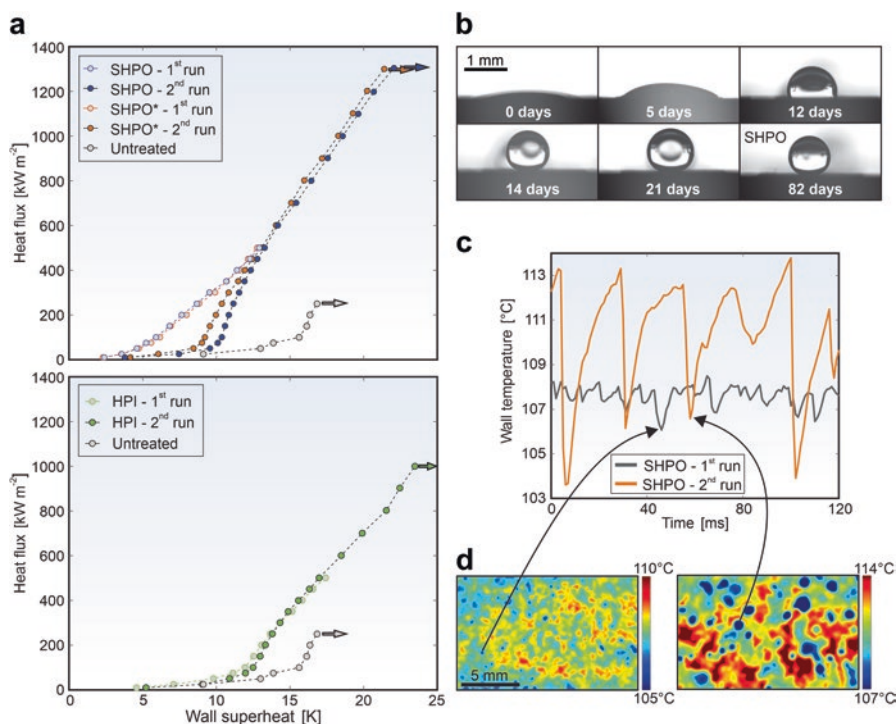


Fig. 23 (a) Pool boiling curves for saturated boiling of water on superhydrophobic and hydrophilic laser-textured surfaces in comparison with untreated stainless steel. (b) An example of naturally developed superhydrophobicity on the SHPO sample under atmospheric conditions (air, 20–25 °C, RH 40–60%). (c) Wall temperature fluctuations underneath an active nucleation site at 200 kW m^{-2} for the first and second boiling run on SHPO sample and (d) corresponding time-averaged temperature fields of the boiling surface

low CHF, matching the values commonly observed in the literature [34, 79, 81]. This Cassie-Baxter boiling regime takes place when the surface structure is initially covered with non-condensable gas (commonly air) or a vapor layer that spreads in all directions as the heat flux is increased, which may be so far the best explanation behind extremely low CHF values reported for most of the superhydrophobic surfaces.

To support the arguments of Shen et al. [199] and Allred et al. [60], we performed additional boiling experiments on a 3×3 array of the laser-textured areas (Fig. 24). High-speed IR thermography and a microthermocouple were used to synchronously monitor the wall and liquid temperature in the vicinity of the active nucleation site. The temperature measurements acquired from the central nucleation area (the left-hand-side of Fig. 24) indicate that bubbles emerge from the non-degassed surface with high periodicity, while the microthermocouple measurement indicates the saturation temperature value (vapor phase temperature) during most of the 0.5-s time frame. This confirms that vapor/gas layer was constantly present on

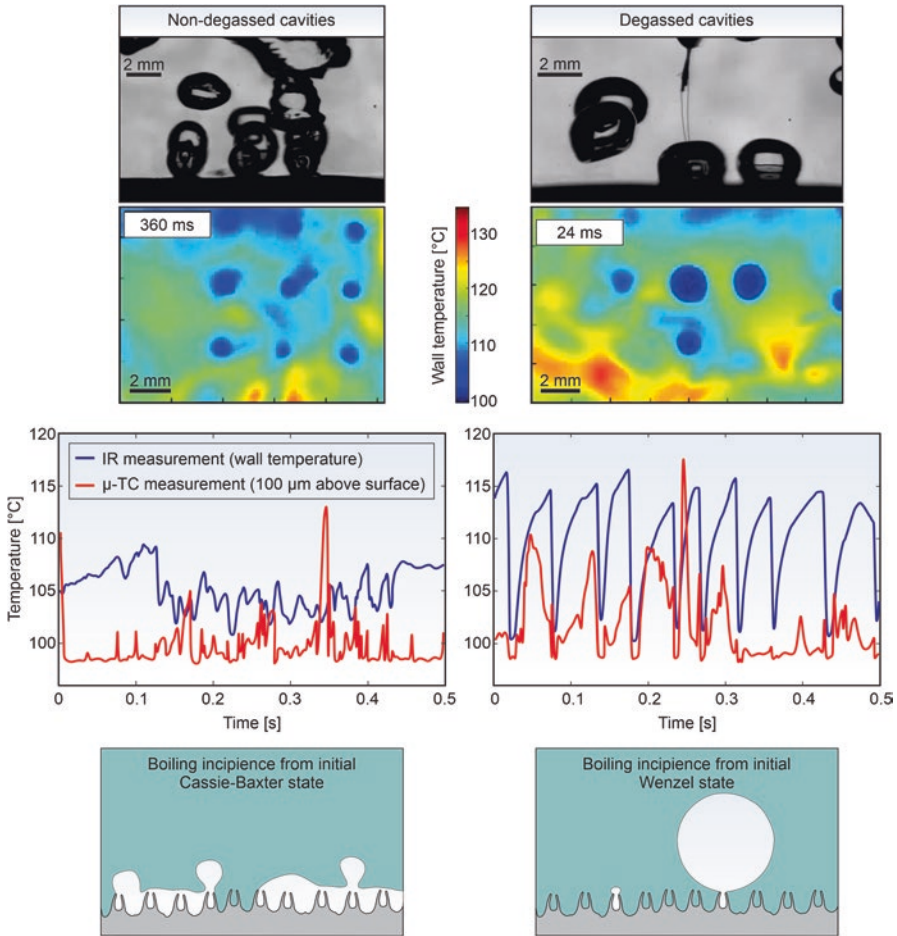


Fig. 24 High-speed images and corresponding temperature fields during the saturated pool boiling of water (pressure slightly below 1 atm) at 100 kW m^{-2} on a stainless steel surface with 3×3 array of the laser-textured areas with μ -cavities for (left) non-degassed and (right) degassed conditions. The size of laser-textured areas is $0.5 \times 0.5 \text{ mm}^2$ and the pitch between them is 3.5 mm. The wall temperature measurements (1 kHz, IR camera) and the liquid temperature measurements (10 kHz, micro-thermocouple placed $100 \mu\text{m}$ above the surface) are provided for the central laser-textured area for a selected 0.5-s time frame. Illustrations below the graphs are showing the bubble growth from μ -cavities in the initial (left) Wenzel and (right) Cassie-Baxter state

the laser-textured areas and provided virtually uninterrupted bubble growth and departure with minimal or no waiting time in between nucleations.

On the other hand, if the surface is successfully degassed either by pressurization, vacuuming or several repeated boiling runs with intermediate condensation of the vapor by slightly subcooling the liquid, the initial Wenzel wetting state is observed. In this situation, as visible from the right-hand side of Fig. 24, the bubble life cycle is initiated by substantial superheating of the surface followed by the

nucleation event and bubble growth (sharp temperature drop associated with high local heat fluxes). After bubble departure, the surface temperature is gradually increased during the waiting time until the nucleation conditions are reached again. The IR thermography proved that non-degassed surface allowed almost simultaneous activation on all nine laser textured areas, which was not observed with the fully degassed surface.

Finally, recent publication by Može et al. [175] convincingly demonstrated that pool boiling of water can be significantly enhanced (in terms of HTC and CHF) on superhydrophobic laser-textured surfaces coated with nanoscale hydrophobic fluorinated silane. Results confirm that the Wenzel wetting regime is possible during boiling on apparently superhydrophobic surface. What is not completely clear, however, is why the vapor layer does not spread around the entire boiling surface at relatively low superheat (i.e. low heat flux) after the boiling incipience. One of the possible reasons is that the underlying micro- and nanostructure, even though covered with hydrophobic layer, prevents the microlayer dry-out and delays the CHF. This would still need to be experimentally confirmed.

The following conclusions can be drawn from the discussed observations and the results reported in the literature [60, 76, 175, 199]:

- Hydrophobic or superhydrophobic surfaces with low receding contact angles benefit from the low-superheat boiling incipience but prevent the vapor spreading. Thus, they do not decrease the critical heat flux. This type of surfaces was termed as parahydrophobic.
- Superhydrophobic surfaces tend to exhibit the Cassie-Baxter boiling regime leading to film boiling immediately after incipience, which is not desirable for practical boiling applications. However, if the boiling on the superhydrophobic surface can be initiated from the Wenzel state, vapor spreading can be suppressed. An important reason behind the Cassie-Baxter boiling behaviour is incomplete degassing of the surface (rather than the liquid alone), which can be avoided in the laboratory environment and has to be seriously considered in practical applications as well.
- The laser-textured surfaces with μ -cavities and time-developed superhydrophobicity show a significant increase in the heat transfer coefficient and maximum attainable heat flux compared to untreated surfaces, which implies the Wenzel boiling behaviour. This is attributed to the appropriate degassing procedure and the fact that superhydrophobicity is not stable during the nucleate boiling. Initially superhydrophobic surfaces showed a static contact angle of $\sim 25^\circ$ after exposure to boiling but still provided lower superheats compared to hydrophilic samples. This proves that (super)hydrophobic properties might still be locally present even after the boiling, which needs a further analytical confirmation.
- Despite the unstable wetting behaviour of laser-textured surfaces, the μ -cavities were shown to enhance bubble nucleation and can provide control over the locations of active nucleation sites regardless of the initial contact angle between the fluid and the surface. This is particularly important for future implementation in real applications.

4.5 *The Influence of Boiling on Surface Chemistry and Wettability*

There are few research papers that discuss the specific changes in surface chemistry and wettability as well as long-term stability of surfaces when used in boiling conditions [49, 200–202]. Despite the importance for real applications, it is presently still not understood well, how newly developed functionalized surfaces are affected by periodic bubble nucleation and associated temperature variations as well as by the relatively high (local) temperatures near the CHF or during the transition to film boiling. In this context, the results by Može et al. [49] are of great importance, since they show the chemical and morphological changes that are induced on the molecular and atomic level due to the critical heat flux incipience as well as the stability of boiling performance on laser-textured copper surfaces in pool boiling of water. It was shown that, similarly as for other materials [46, 62, 84, 175], the laser-textured copper surfaces with μ -cavities enhance the HTC and CHF for 115% and 90%, respectively (Fig. 25a). Moreover, their results convincingly demonstrate better stability (repeatability) of nucleate boiling process on laser-textured surfaces compared to the untreated sample. However, when the surfaces reached CHF and underwent a controlled transition into the film boiling regime with surface temperatures in the range 210–320 °C, the surface experienced a deoxidation through conversion of Cu (II) oxide and hydroxide species into Cu(I) oxide and Cu metal. This was confirmed by high magnification SEM imaging (Fig. 25b) and XPS analysis (Fig. 25c–e).

Changes in surface chemistry and nanostructure constituted the transition from the initial hydrophilic (before reaching CHF) to hydrophobic (after reaching CHF) state, which greatly affected the boiling performance. After exceeding the surface temperature of 200 °C for the first time, the later experiments showed decreased CHF and increased HTC, which corresponds well with the hydrophilic-to-hydrophobic wettability transition.

Contrarily to the observed deoxidation of copper during pool boiling, Trojer et al. [200] intentionally pre-oxidized low-carbon steel samples to simulate the natural oxide layer observed on a reactor pressure vessel wall in light-water reactor nuclear power plants. Oxide layer rendered the surface superhydrophilic and capable to wick water. Flow boiling experiments performed over a wide range of testing conditions proved a consistent increase in the CHF (up to 70%) on pre-oxidized samples compared to the untreated one, which is again in-line with the extreme surface wettability properties. However, to convincingly conclude this CHF enhancement is permanent, additional long-term tests are still required.

Surface wettability and multiscale topography significantly influence the nucleate boiling phenomena. Thus, the stability of the surface from the chemical and topographical perspective will directly affect the boiling performance. Generalization of the long-term boiling performance for numerous combinations of materials,

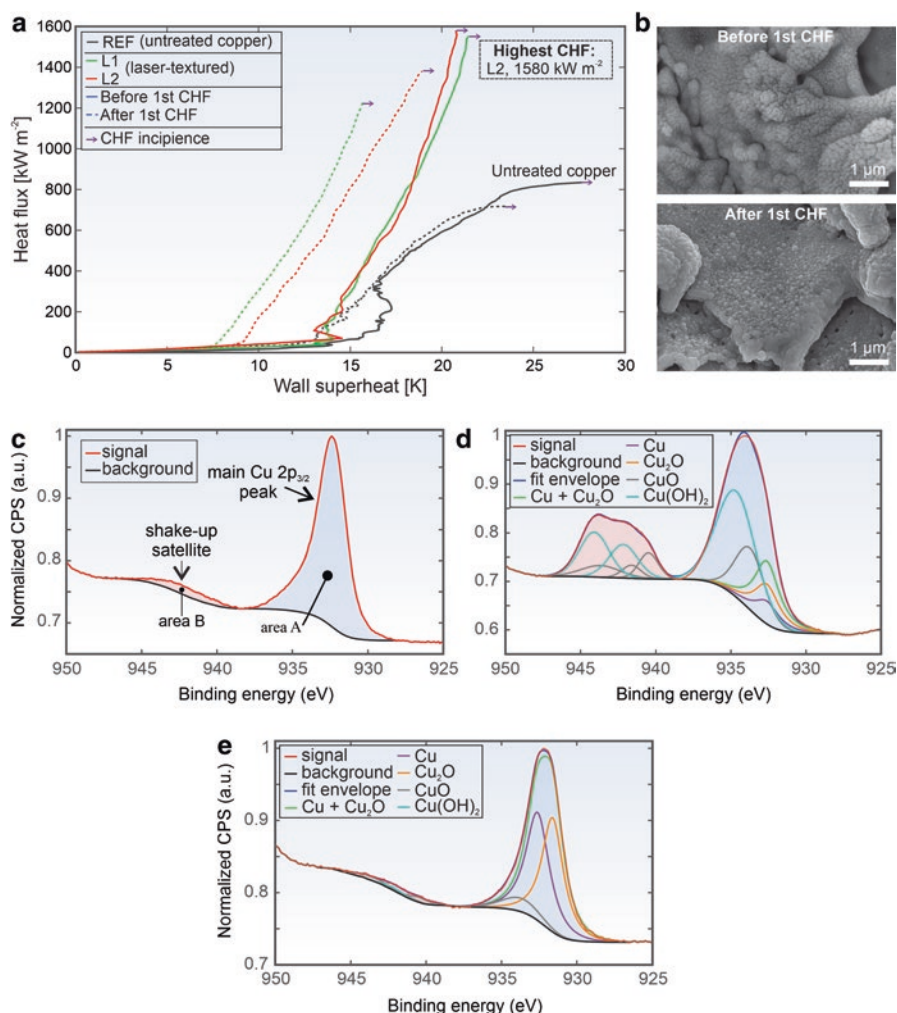


Fig. 25 (a) Pool boiling curves before and after the first incipience of CHF on the untreated and laser-textured copper in air (L1) and argon (L2) atmosphere. (b) SEM images of the L1 sample before and after the first CHF incipience. The narrow XPS scans of the $\text{Cu } 2p_{3/2}$ main peak and its shake-up satellite on the (c) untreated copper surface prior to boiling with additional deconvolution on L1 sample (d) before and (e) after the CHF incipience. (Reproduced and adapted from Ref. [49] with permission from Elsevier)

possible surface engineering methods, working fluids and operating conditions is, however, practically impossible and requires individual studies, which are necessary for later implementation in real phase-change heat transfer applications.

5 Outlook

Undoubtedly, the *surface wettability* influences the nucleate boiling process, but the relation between the boiling performance and wettability is not as straightforward as it looks from a very simplified picture. This commonly used simplified theoretical approach considers solely the preference of the (super)hydrophilic surface to be in the contact with the liquid phase rather than with the vapor phase. Thus, it leads to the conclusion that such surface should delay the attainable critical heat flux, while at the same time it should increase the surface overheat at low fluxes. Conversely, this very simplified view explains that the (super)hydrophobic surface should lower both the temperature of the onset of nucleate boiling and the attainable critical heat flux due to its preference to be in the contact with vapor rather than liquid. The main problem of such simplification is that its validity is limited only to the *ideal* (i.e. chemically homogeneous, chemically stable and completely flat) surface. Unfortunately, ideal surfaces practically do not exist in real applications. Thus, as reviewed in this chapter, neglecting the influence of the surface topography and/or surface chemistry modification during the intense boiling process *inevitably* leads to conclusions that are not consistent with the commonly observed and measured boiling behaviour.

The presented results demonstrate that multiscale surface μ -cavities – when their radii fall within an appropriate range – practically universally enhance the boiling performance with the same surface capable of working well with a various fluids, independent of their polarity, surface tension and heat of vaporization. Moreover, they can ensure stable, controlled and enhanced boiling performance even in combination with binary fluid mixtures. This is of great importance in *real applications*, since diverse boiling systems often use very different working fluids. Additionally, this leads to the stabilization of the boiling process, which is not possible when modified wettability alone is used for surface functionalization. As has been shown, the wettability of the surfaces may change during boiling due to different reasons, including the intensive boiling processes, hydrocarbon contamination and/or ionizing radiation. In this context, it is of great importance that the laser-induced μ -cavities enable enhanced bubble nucleation and provide control over the locations of the active nucleation sites regardless of the contact angle between the fluid and the surface. The ability to produce well-defined areas with different nano-/microtopographies and modified wettability on the micro- and macroscale is of great importance also in *fundamental research*. The unprecedented spatial control over active nucleation sites can be used for the optimization of the active nucleation sites distribution. The results of such studies will, surely, push the heat transfer coefficient and the critical heat flux to even higher values.

Although currently available technologies for surface engineering provide numerous possibilities for their combinations to develop hybrid surfaces with practically any imaginable performance, this usually sacrifices the scalability and increases the complexity as well as the production costs. In this context, *laser surface engineering* represents a straightforward, compact, robust, flexible,

chemical-free and cost-effective alternative. Additionally, laser texturing avoids adding extra surface layers, as is the case when performing surface modification by application of different coatings, and, consequently, such boiling interfaces do not increase the overall thermal resistance of the surface. Due to these reasons, laser surface engineering has the ability to open new possibilities for overcoming the current limitations for further miniaturization of microelectronic devices and has a high potential to increase the performance and safety in high heat flux systems. As always in new emerging technologies, the important still open question remains the *long-term stability* of new types of surfaces for the boiling heat transfer enhancement. This question is of a great importance, since in real applications, the surface and, consequently, boiling stability is measured in years and not in hours as in the everyday scientific experiments.

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