

Thermal Hysteresis in Wettability and the Leidenfrost Phenomenon

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The Leidenfrost temperature (T_L), at which liquid drop lifetime peaks on a superheated surface, is believed to be wettability dependent. Here, we show that the wettability effect on T_L is subject to the history of surface temperature. Observing a water drop evaporating on a polished stainless-steel surface heated from 100–400 °C in argon gas, we find $T_L \approx 265$ °C. We then repeat the experiment along decreasing temperature and find a T_L increase by 10 K i.e., $T_L \approx 275$ °C. This thermal hysteresis is due to reduced contact angle during heating. Once hydrophilized, the hysteresis disappears until the contact angle recovers. Similar observations are made in the air where oxidation is possible.

It was nearly 270 years ago when J. G. Leidenfrost made the careful observation of a drop of water hovering on its own vapor cushion when deposited on a heated spoon [1, 2]. The vapor layer hinders heat transfer from the substrate, resulting in a notably prolonged lifetime of the drop. The Leidenfrost phenomenon, as it is known, continues to attract researchers from various disciplines. This is due to its scientific intrigue complemented by its practical relevance in diverse applications such as cooking [3], metal quenching [4], emergency cooling of nuclear power plants [5], drag reduction [6] and heat engines [7].

The Leidenfrost temperature (T_L)—the surface temperature which maximizes the drop lifetime (τ) plays a pivotal role in the aforementioned applications. Despite the recent progress in theoretical modelling of T_L [8, 9], its precise prediction remains a significant challenge due to various experimental conditions unaccounted for. Among the influential factors including liquid properties [10–15], drop impact [16], ambient pressure [17–20], surface structures [8, 21–26] and surface thermal properties [13, 17, 22], this Letter pays special attention to the effect of wettability. As observed by many, surfaces with a lower contact angle (θ) tend to exhibit higher T_L and vice versa [11, 27–30]. In the context of cooling applications, Nishio and Ohkubo [31] performed mist cooling tests with treated aluminum specimens, revealing that lowering θ resulted in an increase in the minimum film boiling temperature, equivalent to T_L . This trend could be explained, at a fundamental level, by the classical nucleation theory. According to this, lowering θ increases the nucleation energy barrier, limiting the probability of bubble nucleation and subsequent explosive boiling that leads to the Leidenfrost phenomenon [32, 33]. Another possible mechanism for the wettability effect on T_L is vapor film collapse triggered by the liquid-solid van der Waals interaction (intensifies with decreasing θ) [34].

However, does the wettability always remain the same at high temperatures? Tsukamoto et al. noted a drastic decrease in θ from ca. 70° to 0° after quenching stainless-steel plates from 600 °C [35]. Other studies reported wettability enhancement following thermal annealing in argon gas, attributed to the removal of hydrocarbon-based contaminants from the surface [36, 37]. Eventually, the contact angle would recover and become saturated to the original value after a long exposure to the atmosphere. Although Ohkubo and Nishio [31] reported, in fact, similar observations, they still relied on the "saturated" contact angle to infer the wettability effect. Consequently, the impact of surface thermal history on the wettability and its connection to the Leidenfrost phenomenon remain controversial.

In this Letter, we present a comprehensive investigation comprising two sets of experiments. First, we quan-

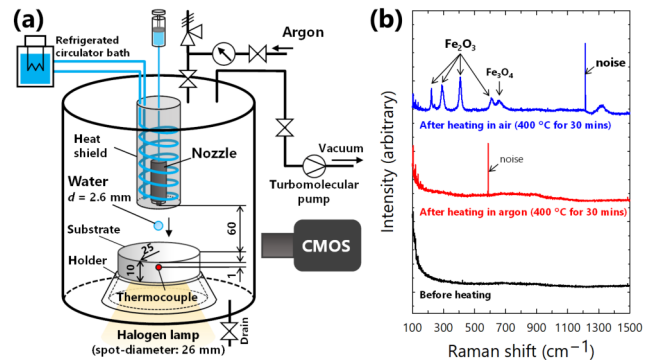


FIG. 1. (a) Experimental setup for drop evaporation on a heated substrate in a controlled environment. (b) Raman spectra obtained from the stainless-steel substrate surface before heating (bottom) and after heating in argon gas (middle) and air (top) at 400 °C for a duration of 30 minutes.

tified the contact angles before/after heating, shedding light on wettability changes at elevated temperatures. Second, we measured the lifetime of a drop (τ) evaporating on the surface at various temperatures. The latter is a rather classical but common method to determine T_L ; in the present work, this was however carried out along both temperature increment and decrement in a carefully controlled environment. Fig. 1 (a) illustrates our experimental configuration comprising a stainless-steel substrate (50 mm diameter \times 10 mm thickness) with a surface roughness of $Ra \approx 10$ nm and a drop dosing system, both housed in a pressure vessel. Substrate temperature was regulated by halogen lamp irradiation from the bottom through a glass window. Visual observation was facilitated by an additional window on the side of the vessel, using a video camera. Prior to each experiment, the vessel underwent initial evacuation to $\sim 10^{-2}$ Pa and subsequent purging with argon (Ar) gas, ensuring a controlled environment free from air exposure and minimizing surface oxidation and airborne contamination. Fig. 1 (b) presents Raman spectra acquired from our substrate before and after heating in various conditions. No apparent changes were observed following heating in argon gas at 400 °C for 30 minutes. In contrast, upon heating in the presence of air, multiple peaks corresponding to iron oxides were detected, albeit with limited visual clarity.

Firstly, the effect of thermal history on wettability is explored. Following a similar approach as outlined in Ref. [31], the substrate (initial $\theta = 57.5^\circ \pm 3.4^\circ$) was initially heated to 150 °C and maintained at this temperature for 30 minutes within an Ar gas environment. Subsequently, the heater was deactivated, allowing the substrate to gradually cool down to room temperature. A drop of deionized water was then carefully deposited on the surface, and the equilibrium contact angle was recorded. This sequence was repeated with elevating the heating target temperature i.e., 200, 250, 300, 350 and 400 °C (see the inset of Fig. 2 for the flowchart). As a result, plotted in Fig. 2, θ showed a noticeable reduction upon heating the substrate above 200 °C, reaching below 10° for the target temperature of 400 °C. Similarly to other works [31, 36, 37], recovery of the contact angle was observed after leaving the substrate in the Ar environment at room temperature: $\theta = 23.8^\circ$ after 1 hour, $\theta = 29.6^\circ$ after 2 hours and $\theta = 48.5^\circ$ after 24 hours. The surface roughness, however, remained unchanged before and after heating i.e., $Ra \approx 10.21$ nm \pm 0.93 nm as measured with the BRUKER DektakXT-E profilometer (the height resolution 0.1 nm), confirming its irrelevance to the contact angle variations. Thus, we conclude that the reduction in θ is primarily caused by the desorption of hydrocarbons.

The amount of hydrocarbons present on the surface is characterized by the surface coverage, denoted by φ , and Oh et al. have demonstrated that θ increases as φ increases [38]. According to adsorption/desorption ki-

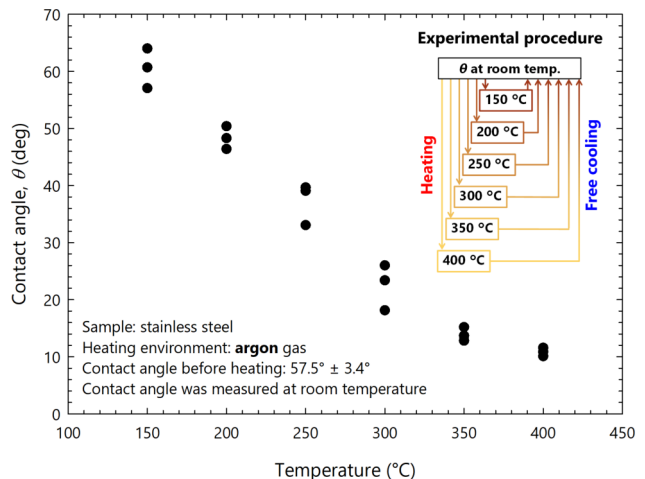


FIG. 2. Contact angles (θ) after heating the substrate to various temperatures in an argon environment. θ was measured three times for each heating condition. The inset is the flowchart of the measurement.

netics, the reduction in φ (and consequently in θ) is subject to the holding temperature and duration. Langmuir demonstrated the existence of an equilibrium surface coverage, φ_{eq} of gas molecules (in our case, hydrocarbons) where the rates of desorption and adsorption are balanced [39]. φ_{eq} decreases with increasing temperature, meaning that the area of hydrocarbon-free surface increases (as described by the Langmuir isotherm). Consequently, a greater reduction in θ is expected at higher holding temperatures, as observed in our results.

As for the effect of heating duration, which was not directly tested in our study, Langmuir also derived an expression for the time (t_{eq}) required to reach φ_{eq} from an initial ϕ and demonstrated that t_{eq} decreases with increasing temperature and vice versa. Thus, shorter heating durations than t_{eq} would result in incomplete removal of hydrocarbons. This could explain why Ohkubo and Nishio [31] observed only a minor or negligible reduction in θ after holding the temperature at 400 °C for 5 minutes. In contrast, we observed a substantial reduction (from ca. 60° to 10°) for the same holding temperature but with a much longer duration of 30 minutes.

Having established the impact of substrate heating on wettability, we now delve into its influence on the initiation of the Leidenfrost phenomenon. As mentioned above, the Leidenfrost phenomenon was characterized via evaporation curves (drop lifetime τ as a function of substrate temperature), obtained as follows. Similarly to the above contact angle experiment, the substrate was carefully heated to 100 °C within an Ar environment. Subsequently, water (density $\rho = 997$ kg/m³, and surface tension $\sigma = 72.8$ mN/m) was steadily fed to the nozzle until it fell in the form of a drop by gravity. The drop was released from 60 mm above the substrate. The resultant

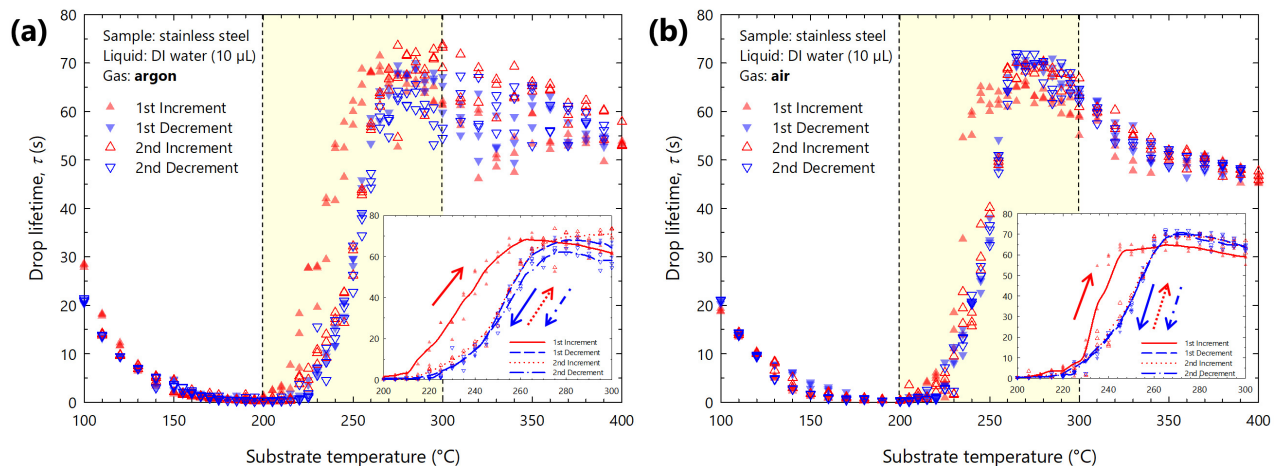


FIG. 3. Evaporation curves presenting the lifetime of a water drop (τ) on a substrate maintained at various temperatures in (a) argon gas and (b) air. The insets show an enlarged view of the data with smooth curves near the transition regime.

drop diameter and impact velocity were approximately $d = 2.6$ mm and $U = 1.08$ m/s, respectively, yielding a Weber number $We = \rho U^2 d / \sigma \approx 44$. To ensure a constant initial temperature of the drop (26.7 ± 0.3 °C), the nozzle was thermally shielded by circulating refrigerated water (see Fig. 1 (a)). Using a video camera, we carefully monitored the evaporation process of the drop and ascertained τ . We then systematically increased the substrate temperature and repeated the experiment. Temperature increments were maintained at 10 K between 100 °C and 150 °C, 5 K between 150 °C and 300 °C, and 10 K up to 400 °C. Following this, we maintained the substrate temperature at 400 °C for a duration of 30 minutes before repeating the experiment in a decremental fashion, stepwisely decreasing the substrate temperature back to 100 °C. As the drops were highly mobile in the Leidenfrost regime, the substrate surface was slightly concaved to prevent the drops from rolling off. To ensure the reproducibility of results, we repeated the evaporation test at least three times for each substrate temperature. However, due to explosive boiling phenomena, especially near the transition to and from the Leidenfrost regime, there were a few instances where the drops experienced splashing beyond the substrate. As a consequence, the volume available for evaporation reduced, leading to significantly shorter τ than the average. We meticulously scrutinized the videos and excluded data points that were clearly influenced by splashing. It is essential to note that the occurrence of this error was minimal (accounting for less than 1% of the total experimental runs).

Our results are plotted in Fig. 3 (a) using red-filled up-triangles and blue-filled down-triangles to represent the increment and decrement runs, respectively. Each case exemplifies the characteristic behavior of drop evaporation on a heated surface: τ decreased as the substrate temperature increased to ca. 220 °C, signifying the nucle-

ate boiling regime. Then, a sharp increase in τ occurred between 220 °C and 280 °C, indicative of the burgeoning vapor layer beneath the drop, profoundly restricting heat transfer and establishing the complete Leidenfrost regime beyond ca. 300 °C. In the decrement case, the trend mirrored the aforementioned observations, albeit in the reverse direction. However, our primary focus lies in the hysteresis—a compelling manifestation of the dependence of τ on the history of the substrate temperature. This hysteresis is clearly evident within the transition regime, as highlighted in Fig. 3 (also enlarged in the inset). Remarkably, the transition regime exhibited an approximately 10 K shift between the increment and decrement cases. Additionally, the Leidenfrost temperatures (T_L), determined as the upper flection point of the evaporation curve, were found to be $T_L \approx 265$ °C for the incremental case whereas $T_L \approx 275$ °C for the decremental one.

This hysteresis and the shift in T_L can be ascribed to the change in wettability induced by substrate heating. Prior to the experiment, the substrate was freshly prepared, and θ was believed to be approximately 60°, although it was challenging to measure accurately due to the surface's concave configuration. As the experiment progressed, the substrate was exposed to elevated temperatures, leading to enhanced hydrophilicity. This augmented wettability facilitated more stable liquid-solid contact at higher temperatures, culminating in the observed higher T_L values. We also note that, in the present study, the change in surface roughness due to heating was negligibly small (~ 1 nm) and had a minimal effect on T_L , compared to others who reported a similar increase in T_L by roughing the surface by ~ 1000 nm [26].

This led us to posit a hypothesis that the temperature hysteresis would cease to exist once the substrate is sufficiently heated to high temperatures. To substantiate

this, we conducted a repeated experiment, engaging in both temperature increment and decrement cycles immediately after the first cycle. The outcomes are illustrated in Fig. 3 (a) as hollow up-triangles and down-triangles for the second increment and decrement cases, respectively, alongside those obtained during the first cycle. As anticipated, the second cycle revealed an absence of temperature hysteresis with all evaporation curves, except for the first increment case, merging altogether. This observation suggests that the saturated contact angle exerts its influence solely before the substrate undergoes high temperatures or after a long exposure to atmosphere to recover the hydrophobicity. From a pragmatic standpoint, it can be inferred that surface wettability plays a minor role in metal quenching, as it typically takes place at high temperatures, rendering the surface sufficiently hydrophilic regardless of the intrinsic nature of the materials.

The temperature hysteresis evident in the evaporation curve is also observable under conditions where surface oxidation is possible. We conducted the same experiment in an air environment, yielding evaporation curves for two temperature cycles (Fig. 3 (b)). As already verified by our Raman spectrum in Fig. 1 (b), the substrate underwent oxidation if not rusting. Nonetheless, the evaporation curves exhibited a similar trend to those in the Ar-environment cases. This implies that the dominant mechanism responsible for the hysteresis is the removal of hydrocarbons from the surface due to heating, rather than oxidation. In comparison to the argon cases (Fig. 3 (a)), one may find relatively small deviations in τ in the Leidenfrost regime (above 300 °C) in Fig. 3 (b). In fact, in the Ar cases, intermittent jumping of the drop was observed, possibly arising from instantaneous contact with the substrate and subsequent explosive boiling, contributing to relatively larger errors. Conversely, the air cases showed more stable hovering of the drops. This disparity could potentially be attributed to unavoidable changes in surface asperities resulting from manual polishing. Another conceivable, albeit unsubstantiated, cause may be the nature of the surrounding gas. Surrounding gases can indeed influence liquid-vapor phase change: prior communications in 1965 discussed the enhanced evaporation rate of water in the presence of gases such as argon and carbon-dioxide [40–42]. These results were attributed to physicochemical effects (e.g., weakening of hydrogen bonding) [43, 44] and/or buoyancy effects (given that argon is heavier than air) [40, 42, 45]. However, it should be noted that their theories were primarily concerned with diffusion-dominant evaporation and may not directly apply to our specific case.

To conclude, our investigation revealed the presence of a temperature hysteresis in the evaporation curves, featuring a notable rise in the Leidenfrost temperature by approximately 10 K following the initial substrate heating. We attributed this to the reduction in contact angle

resulting from high temperature (> 200 °C). The hysteresis was no longer noticeable once the substrate underwent heating. Moreover, we corroborated this phenomenon in the presence of air, establishing that surface oxidation played a negligible role in the observed hysteresis. The understanding of the underlying cause behind wettability changes (i.e., the removal of hydrocarbons from the surface) remains qualitative and is still under debate.

We should also emphasize that the concept of the thermal hysteresis in the Leidenfrost phenomenon, as explored in our study, is distinct from that investigated by Harveys et al. [46] and Chantelot and Lohse [47]. Their research highlighted the existence of temperature gap between the formation and collapse of the Leidenfrost state due to different hydrodynamic mechanisms between them. This type of hysteresis can be observed when the substrate undergoes transient temperature variations while a drop is either in contact with or levitating on the surface. In contrast, our focus centers on the steady-state scenario, with a primary attention toward wettability as the underlying mechanism for the observed hysteresis. However, it is noteworthy that thermally-induced wettability variations may also impact the transient characteristics of the Leidenfrost phenomenon.

Overall, these findings shed light on the intricate interplay between substrate thermal history and wettability, contributing to a deeper understanding of the Leidenfrost phenomenon and its implication in practical applications.

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