

Experimental investigation of Leidenfrost vapor film formation, stability and lifetime on ZnO-based superhydrophobic copper surfaces

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ABSTRACT

In this work, the formation, stability and lifetime of the Leidenfrost vapor film on differently modified copper surfaces are experimentally investigated and the effect of these properties on the reduction of hydrodynamic resistance is examined. The Leidenfrost phenomenon, which manifests itself in the formation of a stable vapor film between a heated surface and a liquid, changes the heat transfer processes and the interaction of the liquid with a solid surface, and therefore, its effect can be applied to reduce hydrodynamic friction.

Four types of copper surfaces were prepared for the study: polished Cu, Cu-FeCl₃, Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS}. Their morphology and crystalline phase composition were evaluated using scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. Surface modification significantly affects the formation of the Leidenfrost vapor film, its stability and lifetime. Experiments were performed with 19 mm diameter pure copper spheres in 20 °C water. It was found that a clearly visible and stable vapor film is formed in water when the copper surface temperature reaches at least 160 °C.

Experiments with free falling samples reveal that different treatments of copper surfaces, along with varying heating boundary conditions, significantly affect the formation of vapor-layer around samples, leading to substantial reductions in drag coefficients. Study gives a valuable insight into the interplay between vapor layer thickness, specimen velocity, and potential applications in fluid dynamics.

1. Introduction

Shipping accounts for about 80% of international trade and is one of the largest sources of fuel consumption in the entire transport industry [1]. During the operation of ships, a large amount of carbon dioxide (CO₂), sulfur (SO_x), nitrogen oxides (NO_x), solid particles, and other greenhouse gases are emitted into the environment. Therefore, the International Maritime Organization (IMO) has set a goal to decrease the total annual greenhouse gas emissions in the shipping sector by 40% by 2030 compared to 2008 [2]. In the maritime sector, fuel consumption is also determined by factors such as the speed of the ship, the performance of the main engine and propeller, the taken route, the position of the ship, and environmental conditions such as wind or current [3].

Investigations also show that rough water conditions significantly increase fuel consumption due to lower hydrodynamic resistance, compared with calm water [4]. Therefore, new methods for reducing environmental impact and achieving better ship energy efficiency have become one of the most important scientific goals.

Findings in scientific literature suggest that ship effectiveness, fuel savings, and environmental harm could be reduced by applying drag reduction. The hydrodynamic drag reduction could be achieved by applying various methods: optimizing ship hulls [5], installing energy-saving devices [6], covering with superhydrophobic antifouling coatings [7], making micro-grooved surfaces [8], constructing air lubrication systems [9], or maintaining a stable air or vapor layer between the object and fluid [10,11]. However, the currently available drag-

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reduction methods have several unavoidable drawbacks, including limited effectiveness [10], high energy demand [12], surface degradation, and instability [13] of the air bubble layer under changing flow conditions. In particular, maintaining a continuous and stable lubricating layer between the solid surface and the surrounding liquid remains a major challenge. In this context, the Leidenfrost phenomenon offers a promising alternative for creating a continuous vapor layer that separates the solid surface from the surrounding liquid, thereby reducing their direct contact and potentially decreasing hydrodynamic drag. Previous investigations have demonstrated that drag reduction under turbulent flow conditions can be achieved using low-temperature Leidenfrost surfaces [14]. The formed vapor layer on the heated surface creates a liquid–vapor interface, which decreases the direct friction through the gas lubrication effect [15]. Studies of vapor layer implementation for drag reduction showed that the vapor layer dramatically affects drag [16]. Leidenfrost temperature (LT) is strongly influenced by surface morphological properties such as wettability and is one of the main parameters defining the beginning of the Leidenfrost phenomenon [17]. Our previous works revealed that the Leidenfrost effect can be applied at low temperatures when working in combination with hydrophobic coatings [18]. Micro/nanostructured surfaces enhance heat transfer compared with non-structured surfaces. The work investigated how to reduce Leidenfrost temperature formation by applying superhydrophobic surfaces (SHS).

SHS can be used not only to accelerate Leidenfrost temperature occurrence on the surface but could also be considered as one of the methods to increase the drag in fluids when applying this layer on a solid material surface. A superhydrophobic surface is defined as a surface having a water contact angle (WCA) above 150° , and a sliding angle (SA) of 10° . Research has shown that when applying a superhydrophobic coating for sphere samples, a reduction in their efficiency can be achieved by 17% and 19% for 20 mm and 30 mm steel spheres, respectively [15]. To improve coating hydrophobic properties, polymer additives such as PDMS (polydimethylsiloxane) [19], PMHS (poly-methylhydrosiloxane) [20] or other additives can be used to change surface morphology. Both polymers increase the hydrophobic film water contact angle and help to maintain coating durability and self-cleaning properties. Investigations have been performed with copper sample plates covered with PDMS polymer and a laser-etched surface, and revealed that in this case, the surface shows excellent properties, with a WCA of 156° and SA of 4.6° , but also withstands a 120-cycle test, which indicates good coating lifetime and durability [21]. Analysis performed with a PDMS polymer additive to hydrophobic coating revealed that coating with WCA of 160.0° and a sliding angle of 2.4° can reduce turbulent drag up to 40% [22]. Another study provides morphological studies performed with TEOS (tetraethyl orthosilicate) application on copper plates and demonstrates that WCA on copper material was in the range 159° – 163.3° (superhydrophobic), while a non-coated copper sample only formed an angle in the range 64.3° – 66.6° [23]. Superhydrophobic surface creation when applying different methods or making several-layer structures shows not only good surface properties but also achieves significant amounts of drag reduction up to 56.8% at low-speed flows when ($Re < 2000$) and around 41.5% when the flow is medium velocity ($2000 < Re < 4000$) [24].

Previous studies have demonstrated that coupling air-bubble injection with hydrophobic surfaces can enhance drag-reduction performance and reduce fuel consumption by approximately 13% [25]. However, the formation, stability, and persistence of thermally generated vapor films on structured superhydrophobic surfaces, particularly under hydrodynamic conditions, remain insufficiently understood. Building on our previous investigation of a ZnO_{PMHS} coating with a water contact angle of 148° [26], the present study examines ZnO tetrapod-modified copper surfaces and evaluates the effects of their morphology and wettability on the Leidenfrost temperature, vapor-film formation, thickness, stability and lifetime, and the resulting hydrodynamic drag. The novelty of this study lies in experimentally establishing

a quantitative relationship between ZnO tetrapod-induced surface structuring, low-temperature Leidenfrost vapor-film behavior, and hydrodynamic resistance. The results demonstrate that combining chemical texturing with a superhydrophobic ZnO_{PMHS} coating promotes stable vapor-film formation at a surface temperature as low as $160^\circ C$ in $20^\circ C$ water, compared with 200 – $220^\circ C$ for the reference surfaces. By correlating vapor-film characteristics with sample velocity and drag coefficient, the study shows that the $Cu-FeCl_3 + ZnO_{PMHS}$ surface achieves an approximately 25% higher average falling velocity than the unheated, untreated copper sample and a drag coefficient as low as 0.39. These findings provide a quantitative experimental basis for the development of surface-assisted Leidenfrost vapor lubrication as a hydrodynamic drag-reduction method. As summarized in Table 1, previous studies have reported Leidenfrost vapor film formation at surface temperatures ranging from approximately 160 to $440^\circ C$, generally under higher water temperatures or different experimental conditions.

The principal strengths of this study are the systematic temperature-dependent characterization of Leidenfrost vapor-film formation on differently modified copper surfaces, the combined application of cooling and free-fall experiments, and the quantitative correlation of surface morphology and wettability with vapor-film characteristics and hydrodynamic parameters. This integrated experimental approach enables the effects of surface modification and temperature on vapor-film formation, sphere velocity, and drag coefficient to be assessed under the investigated laboratory-scale two-phase flow conditions.

2. Materials and methods

2.1. Materials of the specimens

Four different types of copper samples (diameter 19 mm) were selected for the investigation. Copper was chosen due to its durability and high thermal conductivity. The following samples were prepared:

- polished copper (Fig. 1a);
- textured copper ($FeCl_3$ -etched) (Fig. 1b);
- polished copper coated with a TEOS + ZnO + PMHS layer (Fig. 1c);
- copper textured by $FeCl_3$ etching and coated with a TEOS + ZnO + PMHS superhydrophobic layer (Fig. 1d).

2.2. Preparation of Cu samples and surface analysis

2.2.1. Methodology for coating copper samples

B. Preparation of samples for texturing – Polished copper samples (Fig. 1b) were immersed in a 40 wt% $FeCl_3 \cdot 6H_2O$ solution, prepared by dissolving 40 g of $FeCl_3 \cdot 6H_2O$ in 100 g of distilled water. The samples were textured at $45^\circ C$ for 20 min. After texturing, the samples were

Table 1

Comparison of Leidenfrost temperature and drag-reduction performance between the present study and representative literature.

Reference	System/configuration	Leidenfrost vapor film layer temperature	Drag reduction
[14]	Heated rotors immersed in water	surface temperature: 160 – $230^\circ C$, water temperature 86 – $91^\circ C$;	80 – 90% $26000 \leq Re \leq 52000$
[10]	Spheres, model boats	surface temperature: $280^\circ C$, water temperature: $95^\circ C$;	20% decrease in the drag coefficient up to 75%
[27]	Superhydrophobic coating spheres	surface temperature: 260 , 280 , $440^\circ C$, water temperature: 24 , 85 , $95^\circ C$;	
[28]	Heated spheres	surface temperature: $250^\circ C$, water temperature: 85 – $95^\circ C$.	$C_D = 0.04 \pm 0.01$

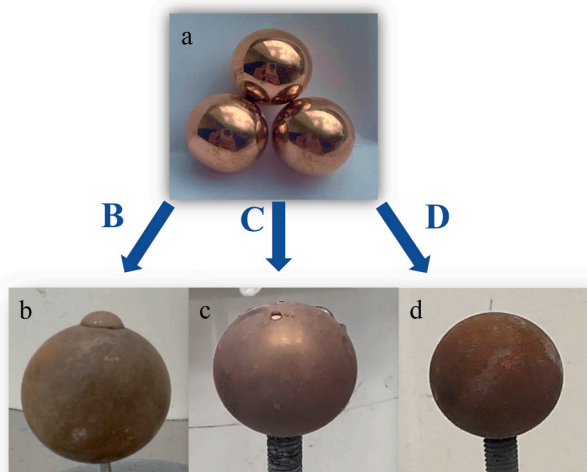


Fig. 1. Differently treated (*B-textured*, *C-coated*, *D-textured + coated*) copper samples: (a) polished copper; (b) copper textured in an FeCl_3 solution; (c) copper coated with a ZnO + PMHS superhydrophobic layer; (d) copper textured in an FeCl_3 solution and coated with a ZnO + PMHS superhydrophobic layer.

removed from the solution, thoroughly rinsed with distilled water, and dried in ambient air at room temperature before further use.

C. Preparation of samples for hydrophobic ZnO + PMHS coating – Polished copper samples (Fig. 1c) are immersed in a 10% TEOS and isopropanol solution with a pH of 3–4 (adjusted with HCl) for about 30 s, then left to dry. The samples are then immersed in a 20 mg/ml ZnO powder in isopropanol solution and left to dry before the next coating step. Then the samples are placed in a 0.5% PMHS isopropanol solution for 30 s. The coated samples were subsequently cured at 100 °C for 1 h in a conventional laboratory oven under ambient air conditions. No inert atmosphere or humidity control was applied during the curing process. The Cassie-Baxter model and earlier research on ZnO nanostructured

surfaces indicate that trapped air can be accommodated in the gaps between ZnO structures, improving water repellency and decreasing the effective solid–liquid contact area [26]. This reduced liquid–solid interaction may facilitate the formation and persistence of a vapor layer at lower surface temperatures (see Fig. 2).

D. Preparation of samples for texturing and hydrophobic coating – Polished copper samples (Fig. 1b) were immersed in a 40 wt% $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ solution, prepared by dissolving 40 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 100 g of distilled water. The samples were textured at 45 °C for 20 min. After texturing, the samples are removed from the solution, rinsed with distilled water, and heated in an oven at 60 °C for 1 h to form an oxide layer suitable for further hydrophobic coating. After cooling, the samples are immersed in TEOS–ZnO–PMHS isopropanol solutions for 30 s. The coated samples were subsequently cured at 100 °C for 1 h in a conventional laboratory oven under ambient air conditions. No inert atmosphere or humidity control was applied during the curing process. The combination of ZnO tetrapods, PMHS polymer, and FeCl_3 etching was selected to achieve a synergistic enhancement of surface roughness and superhydrophobicity. FeCl_3 etching was employed to introduce additional micro-scale surface roughness and surface texture, while the ZnO tetrapods provide hierarchical surface structures and PMHS reduces the surface free energy.

2.2.2. Preparation of TEOS + ZnO + PMHS solutions

C1. Preparation of TEOS solution – Pour 66 ml of isopropanol and 10 ml of TEOS (tetraethyl orthosilicate) into one glass container and stir the solution for about 5 min. In a second glass container, pour 20 ml of isopropanol and 3 ml of acidified deionized water (pH 3–4, acidified with HCl) and stir for 5 min. After mixing, slowly pour the second solution into the first and stir for about 15–30 min.

C2. Preparation of ZnO tetrapod solution – Mix 2 g of ZnO nanoparticles with 100 ml of isopropanol and sonicate for about 15–30 min to ensure uniform dispersion.

C3. Preparation of PMHS solution – Add 0.5 ml of PMHS (poly-methylhydrosiloxane) to 99.5 ml of isopropanol and stir for about 30 min.

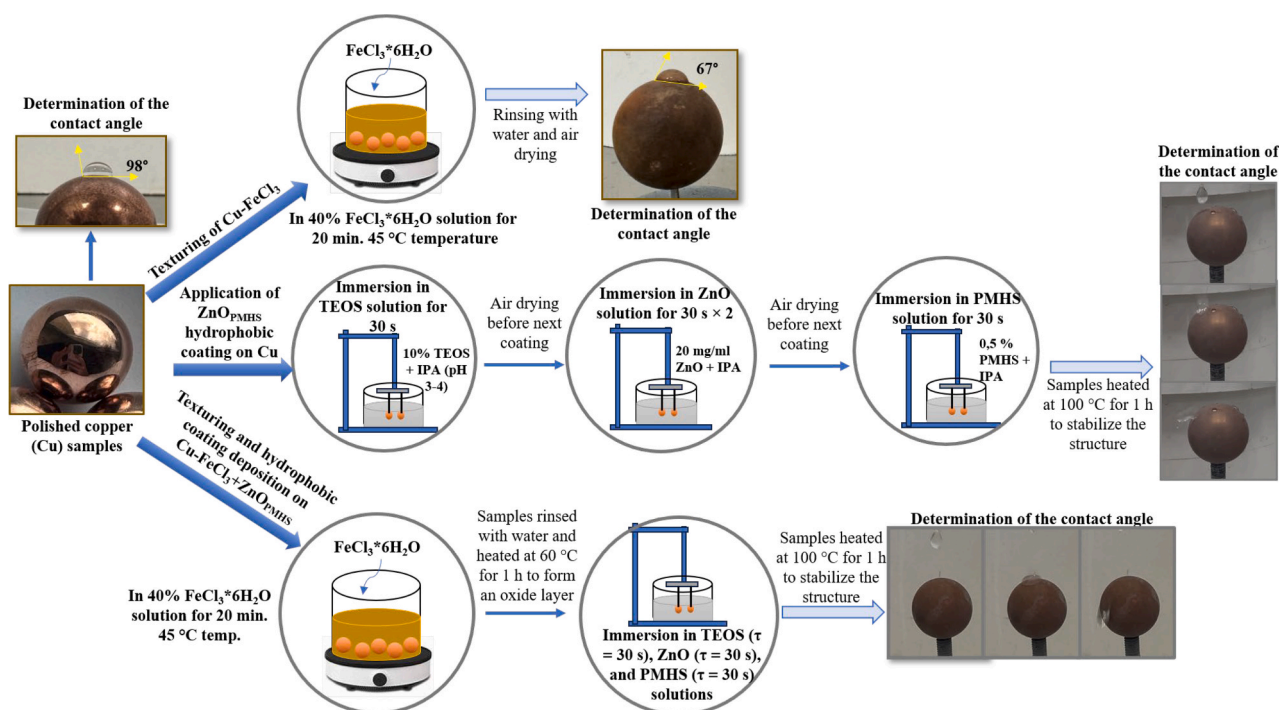


Fig. 2. Methodology for coating copper samples.

2.3. Determination of the hydrophobic contact angle of Cu samples

Before the Leidenfrost effect experiments, the hydrophobicity of the Cu sample surfaces was evaluated. The Young equation was used to assess hydrophobicity by calculating the contact angle between the experimental sample surface and a water droplet of 0,5 ml (Fig. 3).

The water contact angle on the Cu sample surface was 98° , whereas the contact angle of the textured Cu-FeCl₃ sample was 67° . The Cu + ZnOPMHS- and Cu-FeCl₃ + ZnOPMHS-coated samples exhibited excellent superhydrophobic properties. Their contact angle reached $> 150^\circ$, and the impacting water droplet rebounded from the sample surface and was deflected laterally.

2.4. Methodology of SEM analysis

A scanning electron microscope (SEM) uses a focused high-energy electron beam to investigate the sample surface; the beam propagates vertically through the microscope column under vacuum conditions [29]. The electron source is a heated filament that emits electrons, which are subsequently focused and accelerated toward the investigated sample surface by electromagnetic fields.

When interacting with the atoms of the sample, the electrons undergo various interactions, including elastic and inelastic scattering as well as secondary electron emission. These interactions generate signals that are collected by dedicated detectors, enabling the reconstruction of the surface image. To ensure efficient electron-sample interactions, the sample must be electrically conductive or coated with a conductive material, such as a thin gold layer.

By scanning the sample surface with an electron beam and recording the signals at each point, a detailed image of the surface topography and chemical composition is obtained. Owing to its extremely high spatial resolution, reaching the nanometer scale, SEM is one of the most important tools for investigating material morphology and microstructure.

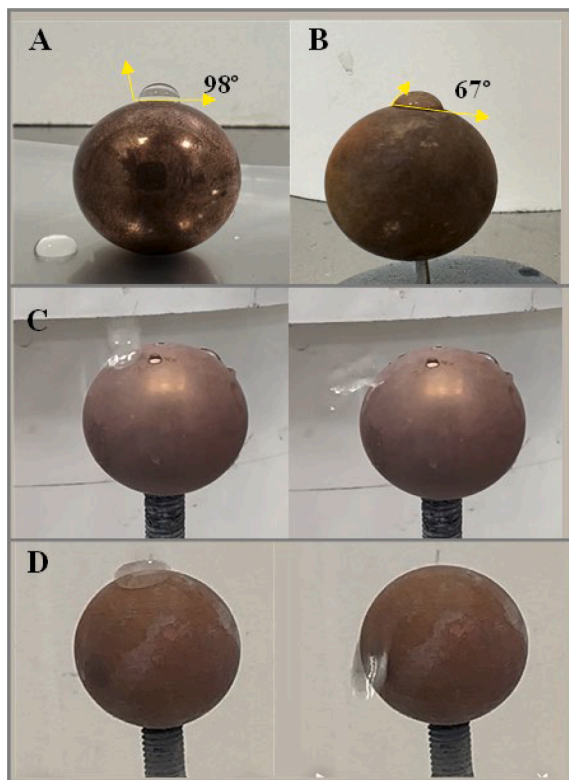


Fig. 3. Water contact angle on A) Cu, B) Cu-FeCl₃, C) Cu + ZnOPMHS, and D) Cu-FeCl₃ + ZnOPMHS surfaces.

2.5. Methodology of SEM-EDS analysis

The morphology and elemental composition of the original and treated metal surfaces were studied using scanning electron microscopy combined with energy-dispersive X-ray spectroscopy. Before analysis, the samples were dried at 40°C and then placed in the microscope chamber. Studies were conducted using a ZEISS EVO MA10 scanning electron microscope at an accelerating voltage of 20 kV. Elemental composition was determined using a Bruker AXS XFlash 6/10 detector. The stated detector accuracy is approximately 1%, and the detection limit reaches 0.1 wt%.

2.6. Methodology of XRD analysis

X-ray diffraction (XRD) analysis followed by Rietveld refinement was performed using a BRUKER D8 ADVANCE diffractometer equipped with Cu K α radiation and operated at an accelerating voltage of 40 kV. The measurements were carried out over a 2θ range from 3° to 70° with a step size of 0.02° . The obtained diffraction data were analyzed using the Diffra. EVA software package.

A separate X-ray diffraction pattern was recorded for each sample type. X-ray analysis was performed over the irradiated surface area of the sample. Therefore, individual point measurements, similar to those in SEM-EDS analysis, were neither performed nor averaged. The central region of the sample, free of visible contamination, mechanical damage, or edge defects, was selected for analysis. Sample positions and scanning conditions were maintained identically for all surfaces studied. This ensured comparability of the diffraction data obtained.

2.7. Investigation of the Leidenfrost process and determination of its critical temperatures

The investigation of the Leidenfrost effect identification and the determination of critical temperatures was carried out in several stages. First, nitrogen gas was introduced into a muffle furnace to protect the investigated samples from oxidation. The copper specimen was internally drilled up to the vicinity of the surface, and a type E thermocouple was inserted into the drilled channel so that its tip was positioned as close as possible to the specimen surface, and a data acquisition system was used to record the cooling process dynamics.

Subsequently, the sample was placed into the furnace and heated to different temperatures (Fig. 4). The investigated samples and experimental conditions are summarized in Table 2.

The temperature range of $160\text{--}280^\circ\text{C}$ was selected based on preliminary observations of vapor film formation on the superhydrophobic ZnOPMHS and FeCl₃ + ZnOPMHS-coated copper surfaces. The lower limit of 160°C corresponds to the experimentally observed onset of vapor film formation on the modified copper surfaces. Therefore, temperatures above this value were selected to systematically investigate the formation, lifetime, and stability of the Leidenfrost vapor film under different thermal conditions. The upper limit was selected based on the observed instability of the vapor film at higher temperatures.

A high-speed optical measurement technique was used to directly detect the vapor film thickness created under Leidenfrost conditions. This technique offers spatially resolved thickness measurements during stable film boiling and permits non-intrusive imaging of vapor film generation. During the Leidenfrost experiments, vapor film formation was identified by the presence of a continuous vapor layer covering the entire heated surface of the sample and separating it from the surrounding water. Vapor film duration was measured from the moment the heated sample first contacted the water surface until the vapor layer collapsed and the sample became fully wet and submerged. The maximum observation period was 20 s. The samples were lowered into the water tank at a constant velocity of 0.01 m/s to a depth of approximately 4 cm below the water surface, ensuring complete submersion of the sample and clear visualization of the vapor film. A Phantom Miro C-

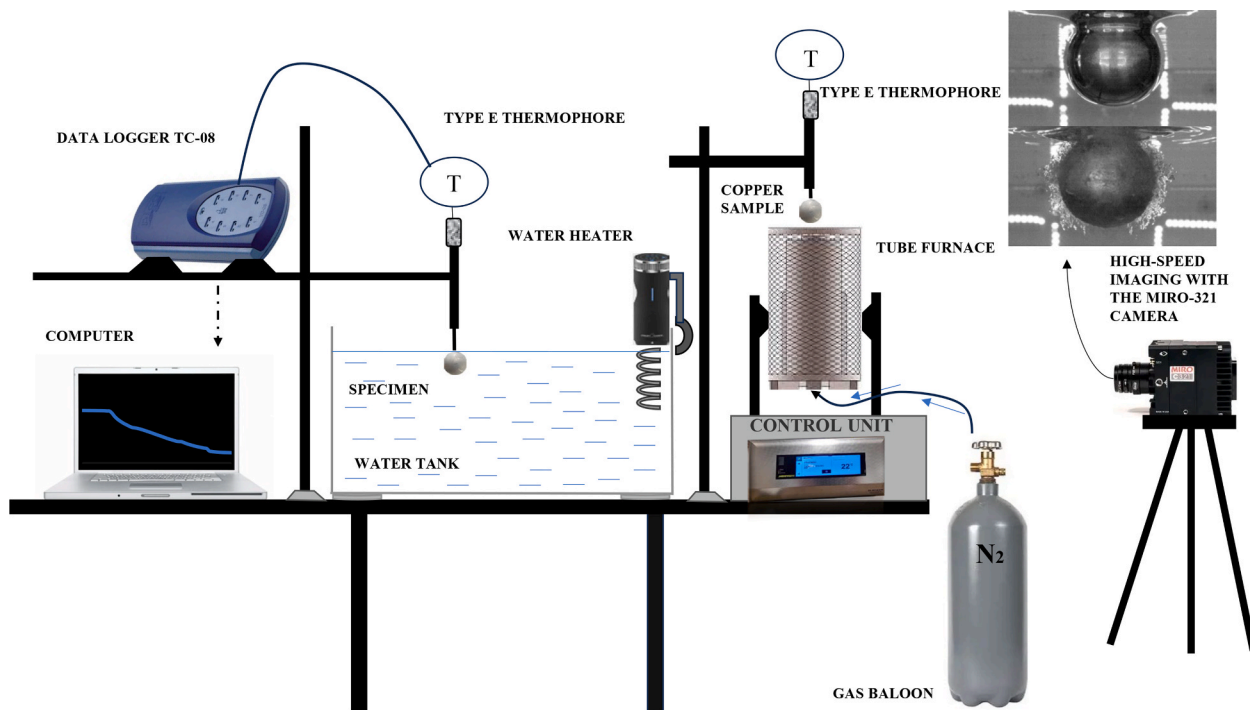


Fig. 4. Experimental setup used to determine the Leidenfrost temperature.

Table 2
Sample types and experimental conditions.

Samples: Cu, Cu-FeCl ₃ , Cu + ZnOPMHS, Cu-FeCl ₃ + ZnOPMHS		Water temperature, °C
Surface temperature °C	160	20
	180	20
	200	20
	220	20
	240	20
	280	20

321 high-speed camera with a resolution of 640×480 pixels and a recording speed of 3290 frames per second was used to measure the thickness of the produced vapor film after the sample and thermocouple reached the required temperature, which was directly monitored using the computer program Data Logging software PicoLog6. For testing, the camera was positioned around 20 cm away from the specimen. The image acquisition parameters were sample rate: 2000, exposure time: 311.34 μ s, and exposure index: 4000. Phantom Video Player, an image processing program, is then used to examine the captured video. The vapor film thickness (in millimeters) is precisely calculated, and the sample in the video is calibrated within the processing program. Before determining the vapor film thickness, the vapor film image was calibrated using the known specimen thickness of 19 mm. The thickness of the vapor layer was determined by measuring the distance between the specimen surface and the outer visible boundary of the vapor film using the calibrated frames. By increasing the vapor layer's visibility in relation to the surrounding area, backlight illumination was used to make the detection of the vapor film border easier. While maintaining sufficient temporal resolution for observing rapid vapor film dynamics, the selected image acquisition parameters reduced motion blur effects during transient operations. The calculated error in vapor film thickness measurement was around ± 0.05 mm. The main sources of uncertainty were pixel resolution, optical aberrations, and the difficulty of accurately determining the vapor film boundary from two-dimensional optical images. The Leidenfrost temperature and vapor-film stability were treated as two separate parameters. It was defined as the lowest tested

temperature at which a continuous vapor film completely covered the immersed sample surface, whereas vapor-film stability was quantified by the duration of continuous film coverage.

To ensure the reliability of the obtained experimental results, all tests were repeated three times under identical conditions. The results revealed that the repeatability of the Leidenfrost effect depends on the copper surface condition and the type of surface treatment. For the polished copper specimen, when the vapor film persisted for up to 1.5 s at a specimen temperature of 240 °C, the deviation of the results was up to 5%. As the temperature increased to 280 °C and the vapor-film lifetime decreased, the deviation increased to approximately 15%. For the textured copper specimen, when a vapor film with a duration of approximately 1 s formed around the specimen, the deviation was approximately 1%. In contrast, when only a minimal vapor film with a duration of approximately 0.5 s was observed, the deviation increased to approximately 17%. For the copper specimen coated with a super-hydrophobic coating, an opposite trend was observed: as the temperature increased and the vapor-film lifetime became longer, the deviation decreased from 13% at 160 °C to less than 1% at 260–280 °C. A similar trend was observed for the textured copper specimen with a super-hydrophobic coating. When the vapor-film lifetime was approximately 3 s at 160–180 °C, the deviation decreased from 15% to less than 1% as the vapor-film lifetime increased to more than 10 s at 200–280 °C. This behavior may be attributed to the instability of short-lived vapor films. When the vapor film persists only for a short period, its formation and stability may vary between individual trials, resulting in greater fluctuations in the measured values and, consequently, larger deviations. In contrast, a longer-lasting and more stable vapor film provides more consistent experimental conditions and therefore improves the repeatability of the measurements.

2.8. Methodology for the determination of hydrodynamic resistance

The experimental setup used in this study was designed to observe the free fall of samples heated in a furnace (Fig. 5). The sample motion was recorded using a high-speed camera (MIRO-C321), and the acquired

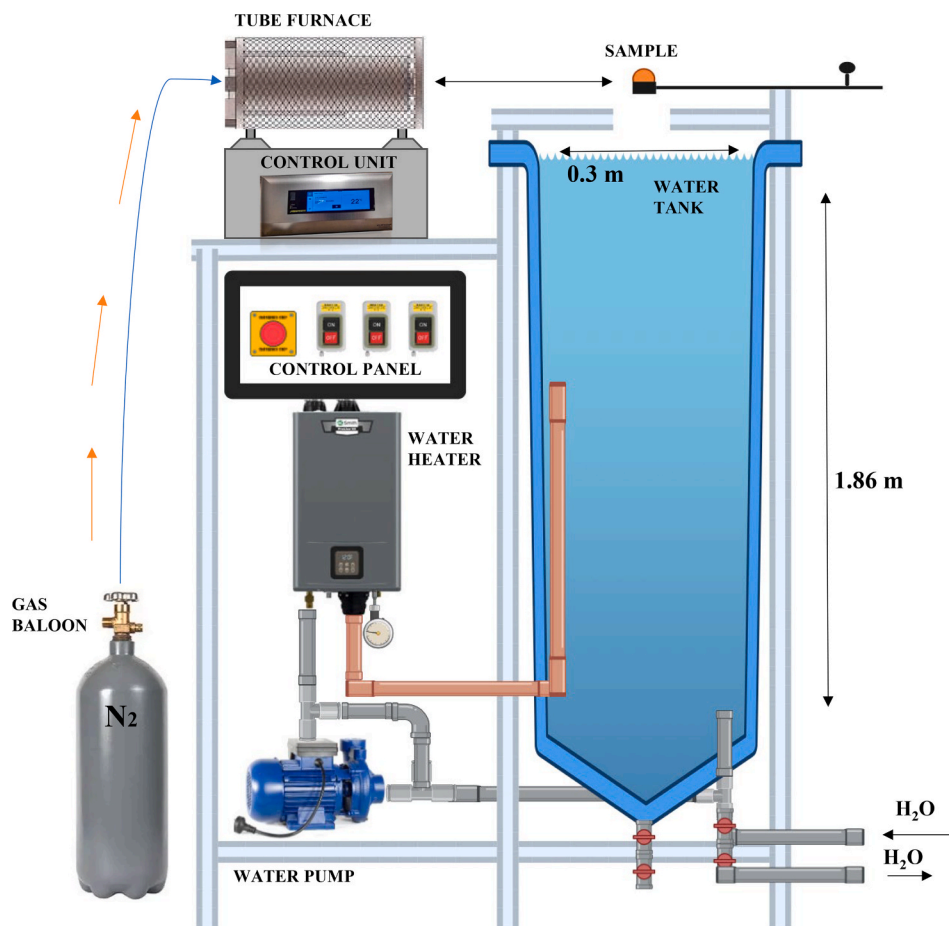


Fig. 5. Experimental setup for hydrodynamic resistance tests.

data were analyzed with the Phantom Video Player software operating under the Phantom Camera Control 3.9 system. A cylindrical water tank with a height of 1.87 m and a capacity of 180 L was used in the experiments. Water was supplied to the tank by opening a valve and heated to 20 °C using a Kospel PPE-9/11/12/15 water heater. Water circulation was ensured by a Stairs Pumps HBI 2-40 RQBE 630 W pump, which also allowed pressure regulation. Nitrogen gas was introduced into the muffle furnace to create an inert atmosphere for the copper samples and to prevent oxidation. During the experiments, free-fall tests were performed to determine the falling velocity of the samples. The heated sample was put in a special holder with a release lever underneath it for the free-fall drag experiments. The lever was turned on once the sample was taken out of the furnace, allowing it to fall freely into the water tank without any further initial disruption or direct mechanical contact. Both cold samples and samples heated in the muffle furnace to temperatures ranging from 160 to 280 °C were released into the water tank. After the fall, the samples were removed through a dedicated extraction valve located at the bottom of the tank. The entire experimental system was controlled via a control panel equipped with an emergency stop button, as well as controllers for the furnace, water heater, and pump. The water tank was divided into three sections, in each of which the falling velocity of the samples was measured, and the dynamic behavior was observed. Each experiment was repeated three times, and the recorded videos were processed using the Phantom Video Player software with the same Phantom Camera Control 3.9 system employed in the previous experimental setup. The same software was also used to calculate the thickness of the vapor film formed during the experiments (mm).

3. Results and discussion

3.1. Morphological analysis of copper sample surfaces using SEM microscopy

Fig. 6 presents the SEM morphological analysis of copper, copper textured in an FeCl_3 solution, copper coated with a polymethylhydrosiloxane polymer, and textured copper coated with a superhydrophobic layer, obtained at 5000 $\times$ magnification. The first image (a) shows the uneven surface relief of the copper plate covered with a copper oxide layer. The copper plate textured with iron (III) chloride (b) exhibits a non-uniform surface on which copper chloride crystals have formed. On the copper surface coated with zinc oxide and a thin polymethylhydrosiloxane polymer layer (c), which together form a superhydrophobic coating, a layer of ZnO tetrapods is observed, preventing direct contact between water and the sample surface. The final image (d) shows a textured copper plate coated with a superhydrophobic layer, characterized by a rough surface formed during texturing, which is additionally covered with zinc oxide tetrapods and a polymer layer.

3.2. SEM-EDS analysis

SEM-EDS analysis (Fig. 7) was performed at 12 measurement points, three points on each sample. The points were selected in spatially separated areas of the studied surface. The characteristic morphological features of each sample were taken into account. Edge areas, visible contaminants, and defects arising during sample preparation were excluded from the analysis. The elemental composition values presented

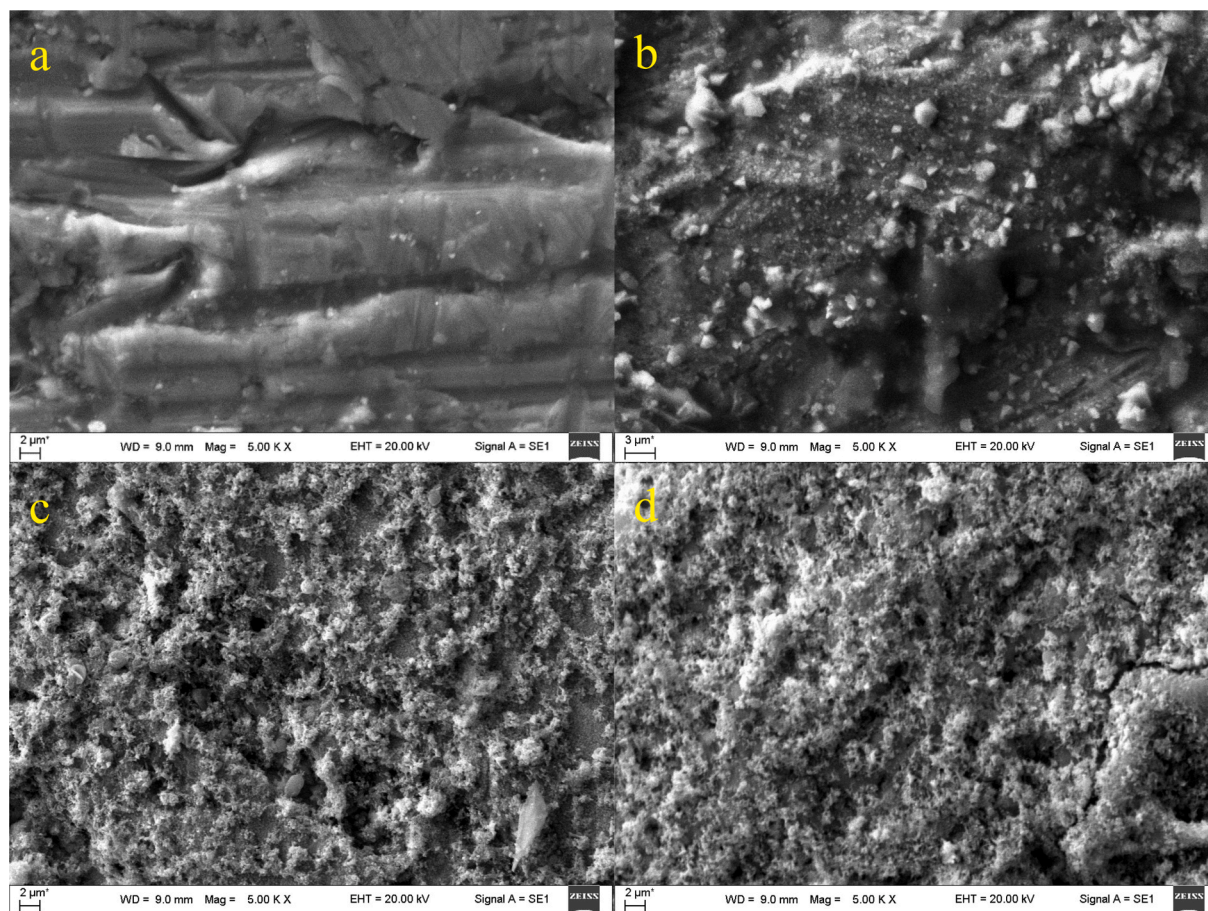


Fig. 6. SEM images of (a) polished copper, (b) copper textured in an FeCl_3 solution, (c) copper coated with a superhydrophobic ZnO_{PMHS} layer, and (d) copper textured in an FeCl_3 solution and coated with a superhydrophobic PMHS layer.

are the arithmetic means of three measurements performed for each sample. This approach made it possible to account for the heterogeneity in the elemental composition of the treated surfaces.

Sample 1 was dominated by copper (72.92 wt%), carbon (20.12 wt%), oxygen (6.27 wt%) and a small amount of aluminium (0.69 wt%). Sample 2 showed a nearly equal distribution of copper (38.01%) and carbon (35.30%), together with oxygen (12.14%), chlorine (9.75%), and trace amounts of silicon and aluminum. Sample 3 contained high concentrations of copper (52.05%), oxygen (15.63%), and zinc (15.11%), in addition to carbon (8.30%), while silicon, sodium, chlorine, and aluminum were detected in trace amounts. In sample 4, the most abundant elements were copper (31.83%), oxygen (21.97%), and zinc (15.25%), followed by chlorine (11.61%) and carbon (9.07%).

3.3. XRD analysis

X-ray diffraction (XRD) analysis was used to identify the crystalline phases present in the copper plates after different surface treatments, as shown in Fig. 8. The pristine Cu sample exhibited diffraction peaks characteristic of metallic copper. After treatment with FeCl_3 , additional reflections were observed in the Cu- FeCl_3 sample. According to phase analysis, these reflections were assigned to CuCl and $\text{Cu}_2\text{Cl}(\text{OH})_3$. No iron-containing crystalline phases were detected. The Cu + ZnOPMHS sample exhibited weak reflections corresponding to crystalline ZnO. The ZnO content of this sample was quantified at 0.76 wt%. The Cu- FeCl_3 + ZnOPMHS sample contained 0.68 wt% ZnO, as well as CuCl and $\text{Cu}_2\text{Cl}(\text{OH})_3$ phases. Since the ZnO content in both coated samples was low and close to the detection limit, these results should be interpreted with

caution. They provide only limited evidence of the presence of a minor crystalline ZnO phase. X-ray diffraction data do not confirm the presence of a chemical bond between the coating and the copper substrate, interaction between ZnO and the PMHS matrix, or surface passivation.

Thus, the XRD patterns were dominated by signals from the copper substrate. CuCl and $\text{Cu}_2\text{Cl}(\text{OH})_3$ phases were identified after treatment with FeCl_3 . The ZnO content was 0.76 wt% in the Cu + ZnOPMHS sample and 0.68 wt% in the Cu- FeCl_3 + ZnOPMHS sample. Since both values are close to the detection limit, XRD provides only limited evidence of the presence of crystalline ZnO. These data do not establish a chemical bond between the coating and the copper substrate or confirm surface passivation. The presence and morphology of the coating are discussed separately based on SEM observations.

3.4. Analysis of thermochemical processes occurring in water on Cu samples at different sample temperatures

In previous studies, aluminum samples coated with a ZnO layer combined with a PDMS (polydimethylsiloxane) polymer were investigated. It was found that the superhydrophobic coating extended the vapor film residence time by a factor of 3.5 and reduced the Leidenfrost temperature from 300 to 200 °C [30]. Therefore, in the present study, the samples were also modified by applying a superhydrophobic coating based on ZnO and PMHS (polymethylhydrosiloxane) to enhance vapor film formation and stability.

In this study, copper samples (19 mm in diameter) were selected due to their higher thermal conductivity compared to aluminum, which allows vapor film formation at lower temperatures. In addition, copper is

more durable and exhibits a longer service life than aluminum.

The investigation of the Leidenfrost process on copper-based samples was carried out using an experimental setup designed for Leidenfrost temperature determination (Fig. 4). To determine the optimal temperature for the onset of the Leidenfrost effect, experiments were conducted at different copper sample temperatures, increased in increments of 20 °C. Due to the relatively low heat capacity of copper (380 J/(K·mol)), the lowest temperature at which a vapor film formed around the sample was 160 °C, while the highest investigated temperature was 280 °C. Experiments were performed using Cu, Cu-FeCl₃, Cu + ZnO_{PMHS}, and Cu-FeCl₃ + ZnO_{PMHS} samples, with the water temperature maintained at 20 °C (Fig. 9).

When the copper samples were heated to 160 °C, it was observed that neither the polished Cu nor the textured Cu-FeCl₃ samples formed a

vapor film. Consequently, the temperature change in the cooling curves was clearly distinguishable when compared to the superhydrophobic Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples, which, under the same conditions, formed a sufficiently thick vapor film that persisted on the sample surface for approximately 3 s and gradually thinned. This behavior indicates that surface texturing and superhydrophobic coating not only promote vapor film formation but also significantly extend its lifetime, thereby reducing the rate of heat transfer from the sample to the surrounding liquid compared to uncoated copper.

At a sample temperature of 180 °C, the polished copper sample did not form a vapor film, whereas the textured Cu-FeCl₃ sample formed a vapor film lasting approximately 1 s. Vapor films were also observed for the Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples, with lifetimes of up to 5 s.

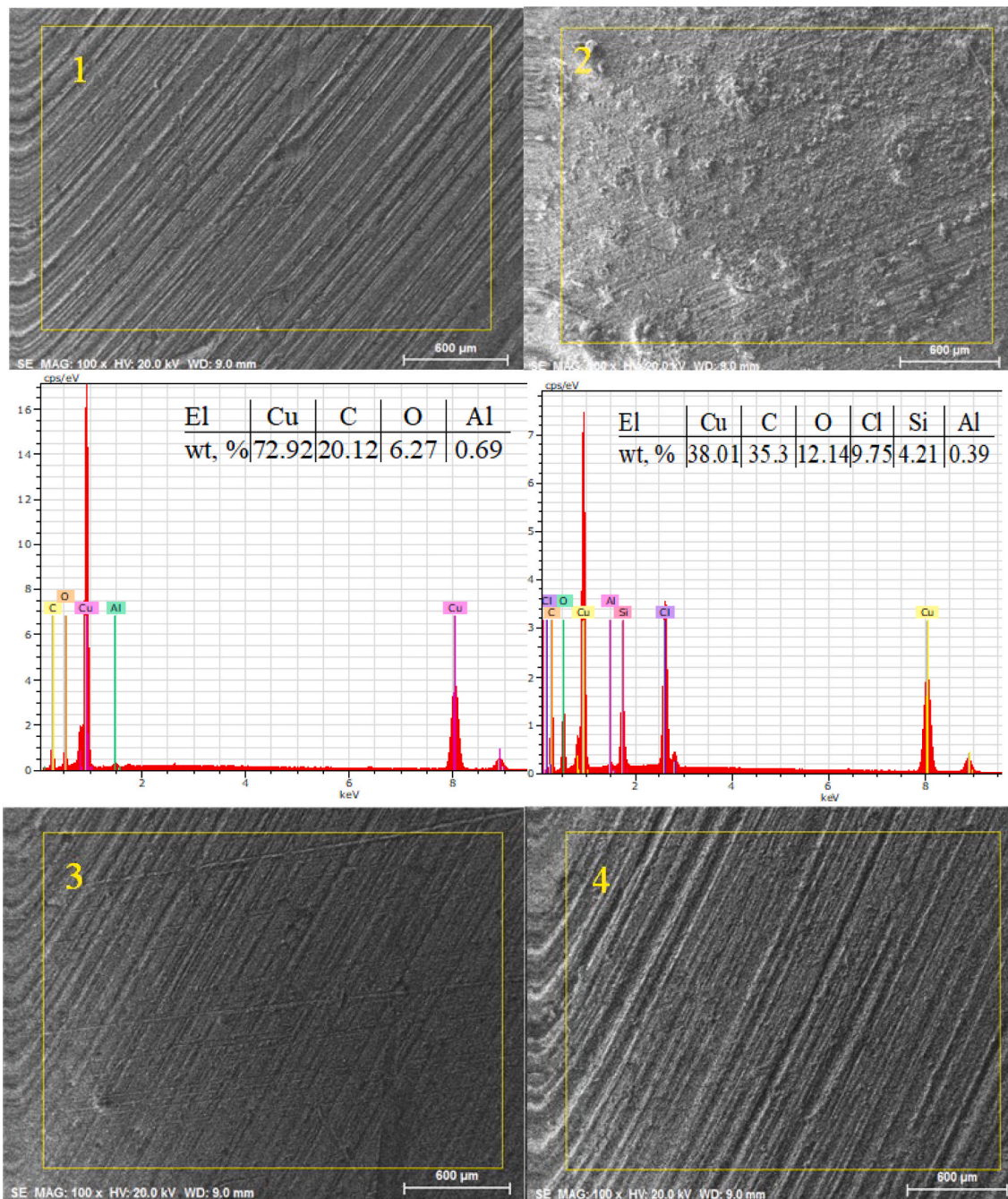


Fig. 7. SEM-EDS analysis performed on 1) a copper plate, 2) a copper plate textured with FeCl₃·6H₂O, 3) a copper plate coated with a superhydrophobic ZnO_{PMHS} layer, and 4) a copper plate textured with FeCl₃·6H₂O and coated with a superhydrophobic ZnO_{PMHS} layer.

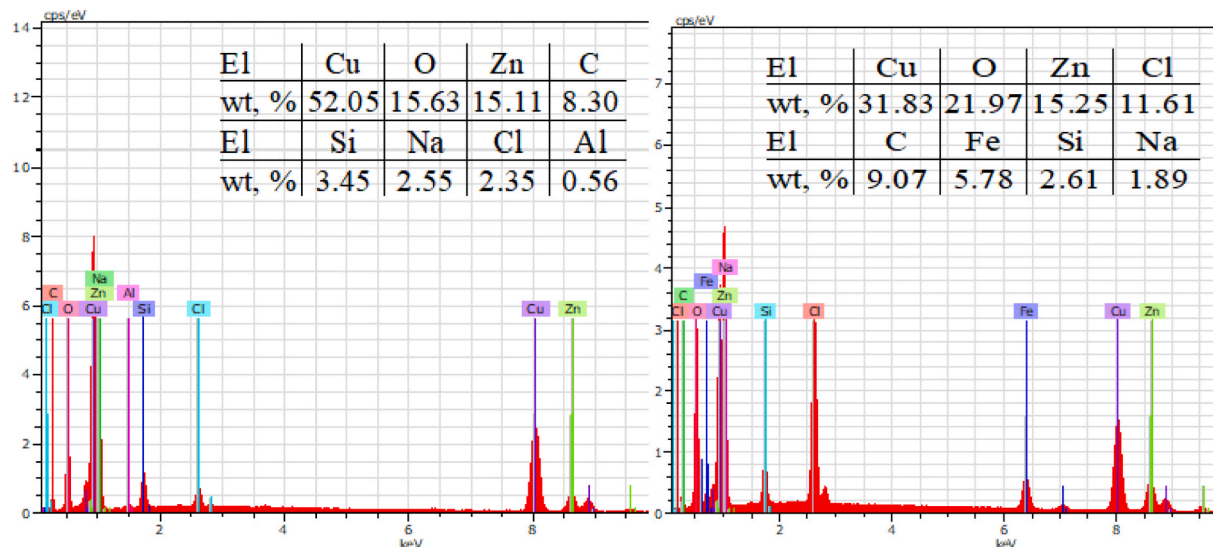


Fig. 7. (continued).

When the sample temperature reached 200 °C, polished copper still did not form a vapor film, while textured copper formed a vapor film lasting up to 2 s. The Cu + ZnO_{PMHS} sample formed a vapor film lasting approximately 5 s, whereas the Cu-FeCl₃ + ZnO_{PMHS} sample exhibited a vapor film persisting for more than 10 s and gradually thinning.

At 220 °C, the polished copper sample formed a thin vapor film (Table 3) with a thickness of 0.3951 mm, which persisted for approximately 1 s before collapsing. The Cu-FeCl₃ textured sample also formed a vapor film lasting about 1 s; however, the film was thicker. In contrast, the Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples formed thick vapor films that persisted for more than 10 s.

At a sample temperature of 240 °C, polished copper formed a thicker vapor film lasting approximately 1 s, whereas the textured copper formed a vapor film lasting only about 0.5 s. As shown in Fig. 9, this resulted in a sharp temperature drop due to the formation of a very thin vapor film (0.3146 mm). Under the same conditions, the Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples formed thick vapor films that persisted for more than 10 s; however, the Cu-FeCl₃ + ZnO_{PMHS} sample produced a slightly thicker vapor film, resulting in a longer cooling duration.

When the sample temperature was increased to 260 °C, both the Cu and Cu-FeCl₃ samples formed vapor films lasting approximately 1 s, with the film formed on textured copper being thicker (Table 3). In contrast, the Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples formed the thickest vapor films, persisting for more than 10 s, with measured thicknesses of 0.8778 mm and 0.8915 mm, respectively.

At the highest investigated temperature of 280 °C, corresponding to the upper limit for stable Leidenfrost effect formation, the vapor film was observed to thin and become more difficult to sustain, regardless of the surface type. During the experiment, the polished copper sample formed a vapor film lasting approximately 1 s, while the textured copper sample did not form a vapor film. At this temperature, the textured Cu-FeCl₃ surface exhibited enhanced heat transfer due to its increased effective surface area, preventing the vapor film from forming and stabilizing. The Cu + ZnO_{PMHS} sample formed a vapor film lasting about 5 s, whereas the Cu-FeCl₃ + ZnO_{PMHS} sample formed a thick vapor film that gradually thinned and persisted for more than 10 s.

The vapor film lifetime and thickness determined for all investigated samples are summarized in Tables 3.

Table 3 presents the thickness of the vapor film formed around the samples, expressed in millimeters. During the study, it was observed that the thinnest vapor films were formed on polished copper samples, particularly at sample temperatures of 220 °C (0.3951 mm) and 280 °C (0.3425 mm).

Textured copper, although exhibiting a vapor film lifetime similar to that of polished copper, formed a thicker vapor film (except at 240 °C). This behavior is also reflected in the cooling curves (Fig. 9), where a less abrupt temperature drop is observed compared to the Cu samples.

The Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples showed similar trends in vapor film lifetime; however, the Cu + ZnO_{PMHS} samples were found to form exceptionally thick vapor films, ranging from 0.775 to 0.889 mm, depending on the sample temperature. The Cu-FeCl₃ + ZnO_{PMHS} samples formed the longest-lasting vapor films in the temperature range of 200–280 °C and also exhibited the greatest vapor film thickness at a sample temperature of 240 °C, reaching 0.891 mm.

Lowering the Leidenfrost temperature is technically significant because a stable vapor film can be established without heating the surface to the higher temperatures typically required for bare, polished copper. This may reduce the thermal load imposed on the substrate and coating and may potentially decrease the energy input required to establish and maintain the vapor-film regime. In addition, operating at a lower surface temperature can broaden the practical applicability of Leidenfrost-based drag-reduction approaches, particularly in applications where maintaining very high surface temperatures is undesirable or technically difficult.

3.5. Calibration of sample surface temperature and determination of heating time

Since the surface temperature of spherical samples cannot be measured directly, surface temperature calibration was required, i.e., determination of the heating time needed to reach the target surface temperatures. For the experiments, a hole was drilled in each sample, into which a Type E thermocouple was inserted and connected to a data logger that transmitted the data to a computer running PicoLog software. Measurement accuracy was ensured by repeating the heating-time measurements several times. The collected data allowed precise determination of the time required for the surfaces of the Cu, Cu-FeCl₃, Cu + ZnO_{PMHS}, and Cu-FeCl₃ + ZnO_{PMHS} samples to reach the target temperatures of 160, 180, 200, 220, 240, 260, and 280 °C. During the experiments, the furnace temperature was consistently set 100 °C higher than the desired sample surface temperature. To create an inert environment, nitrogen gas was supplied to the furnace throughout the heating process. Based on the obtained results, the average heating time for the samples was determined.

The time required for the copper samples to reach a temperature of 160 °C ranged from 6:30 to 7:40 min; for 180 °C, from 7:00 to 10:15 min;

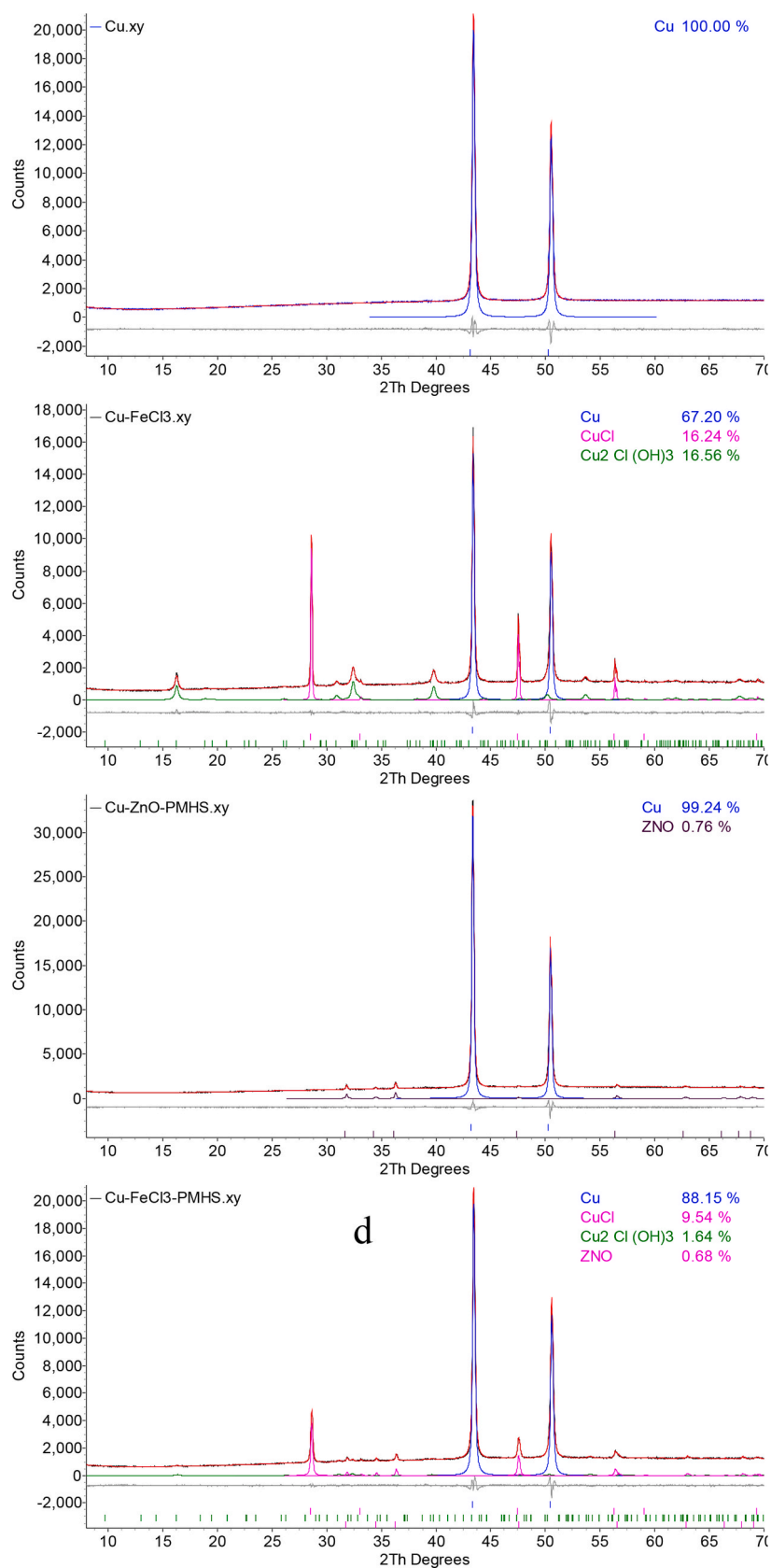


Fig. 8. XRD patterns of a) copper, b) copper textured in an FeCl₃ solution, c) copper coated with a superhydrophobic ZnO_{PMHS} layer, and d) copper textured in an FeCl₃ solution and coated with a superhydrophobic PMHS layer.

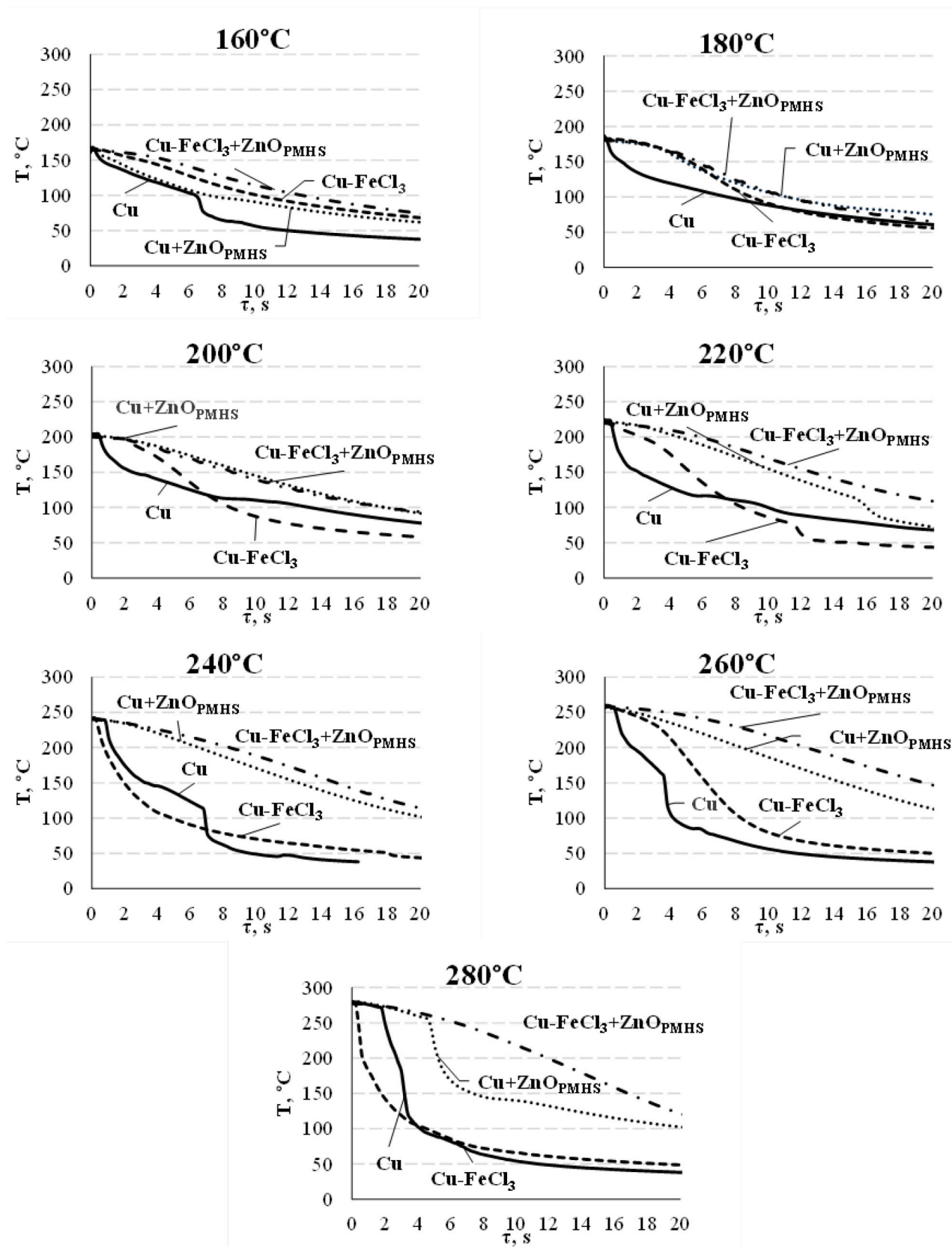


Fig. 9. Cooling dynamics of copper samples with different surface conditions in water at 20 °C.

Table 3
Lifetime and thickness of the vapor film formed on copper samples with different surface conditions.

Sample surface temperature (°C)	Cu		Cu-FeCl ₃		Cu + ZnO _{PMHS}		Cu-FeCl ₃ + ZnO _{PMHS}	
	Lifetime of vapor film (s)	Thickness of the vapor film (mm)	Lifetime of vapor film (s)	Thickness of the vapor film (mm)	Lifetime of vapor film (s)	Thickness of the vapor film (mm)	Lifetime of vapor film (s)	Thickness of the vapor film (mm)
160	no film formed	no film formed	no film formed	no film formed	1–2	0.775	3	0.661
180	no film formed	no film formed	1	0.637	5	0.779	3	0.661
200	no film formed	no film formed	1–2	0.757	5	0.881	>10	0.775
220	1.5	0.395	0.5	0.494	>10	0.887	>10	0.783
240	1	0.668	0.5	0.314	>10	0.878	>10	0.891
260	1	0.596	1	0.798	>10	0.876	>10	0.867
280	1	0.342	no film formed	no film formed	5	0.872	>10	0.781

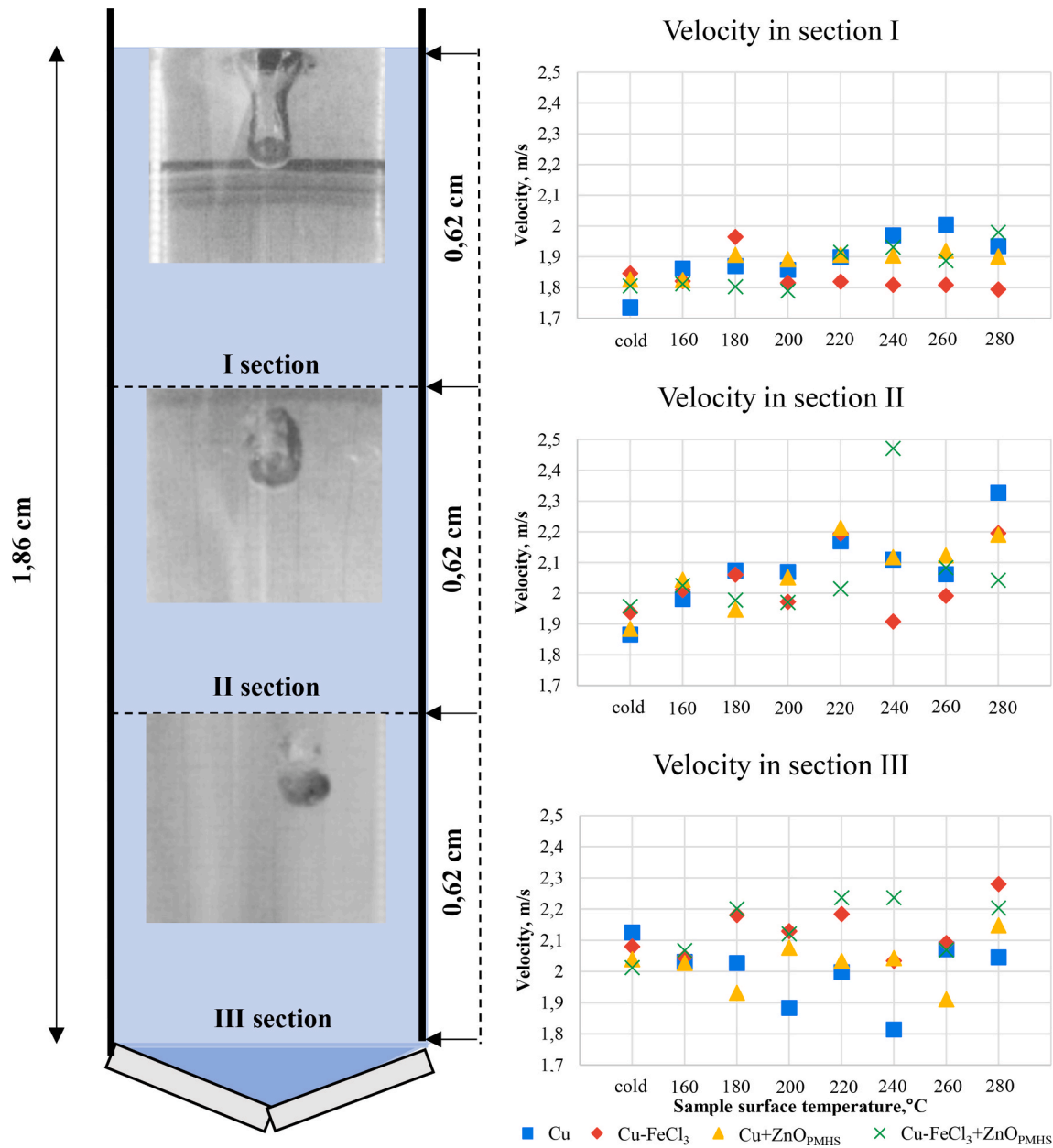


Fig. 10. Hydrodynamic resistance investigation in water at 20 °C using differently treated copper samples: Cu, Cu-FeCl₃, Cu + ZnO_{PMHS}, and Cu-FeCl₃ + ZnO_{PMHS}.

for 200 °C, from 7:40 to 9:15 min; for 220 °C, from 8:30 to 11:40 min; for 240 °C, from 8:30 to 10:20 min; for 260 °C, from 8:40 to 11:20 min; and for 280 °C, from 8:30 to 11:00 min.

3.6. Hydrodynamic resistance investigation of Cu samples in water at 20 °C

Hydrodynamic drag experiments with Cu samples were carried out using the same experimental setup designed for determining hydrodynamic resistance (Fig. 5). For result evaluation, the water column was divided into three equal sections of 62 cm each, to calculate the falling velocity of the samples at different locations of the cylinder and to assess the influence of the formed vapor film on the falling velocity (Fig. 10). Four different types of copper samples were investigated: Cu, Cu-FeCl₃, Cu + ZnOPMHS, and Cu-FeCl₃ + ZnOPMHS. The experiments were divided into four distinct regimes: cold-state baseline, low-temperature regime, medium-temperature regime, and high-temperature regime.

3.6.1. Cold-state baseline

Before heating, hydrodynamic resistance measurements were performed with unheated copper samples to evaluate the effect of temperature on the falling velocity of samples with different surface conditions (Fig. 10). In the first section of the cylinder, the Cu sample exhibited the lowest falling velocity (1.735 m/s) due to strong interaction with the water surface and surface tension forces, which increased hydrodynamic resistance at the initial stage of motion. In the second section, the sample began to accelerate and reached 1.865 m/s, and in the third section – 2.125 m/s, indicating that the falling velocity did not reach a steady state along the entire channel length and gradually approached free-fall conditions.

A similar trend was observed for the textured Cu-FeCl₃ sample. Due to the rough surface, cavities were filled with water, reducing the effect of surface tension forces compared to polished copper. As a result, in the first section, the falling velocity increased by ~ 6.4% (from 1.735 to 1.846 m/s), indicating reduced hydrodynamic resistance compared to polished copper. In the second section, the sample accelerated to 1.938 m/s, and in the third section reached 2.080 m/s.

The Cu + ZnOPMHS and Cu-FeCl₃ + ZnOPMHS samples exhibited lower falling velocities in the first section – 1.826 m/s and 1.805 m/s, respectively. This is attributed to the air layer formed around the sample, which acts as an additional buoyant force opposing the direction of fall for the formed LE buoyant forces are dominant versus gravity forces), thereby increasing overall hydrodynamic resistance and reducing the falling velocity. In the second section, the Cu + ZnOPMHS sample accelerated to 1.884 m/s, while the Cu-FeCl₃ + ZnOPMHS sample reached 1.957 m/s. In the third section, both samples reached their maximum velocities: 2.039 m/s and 2.012 m/s.

3.6.2. Low-temperature regime (160–180 °C)

When the temperature of the Cu sample was increased to 160 °C, the falling velocity along the entire channel length did not stabilize: it reached 1.862 m/s in the first section, 1.981 m/s in the second, and 2.031 m/s in the third. In this case, a vapor film had not yet formed; however, the increased surface temperature reduced the fluid viscosity near the surface and improved flow around the sample, thereby reducing hydrodynamic resistance and increasing the falling velocity. The textured Cu-FeCl₃ sample exhibited a similar trend (1.821 → 2.045 m/s). The superhydrophobic-coated sample (Cu + ZnOPMHS) showed the lowest velocity in the first section of the channel (1.824 m/s), as the formed vapor/air film created additional resistance. In the second section, after the vapor film detached from the sample, the velocity increased to 2.044 m/s, and in the third section it stabilized at 2.027 m/s. The textured and superhydrophobic-coated Cu-FeCl₃ + ZnOPMHS sample exhibited the lowest velocity in the first section (1.812 m/s), as a stable vapor film of approximately 0.661 mm thickness enhanced resistance when buoyant forces overcome gravity forces and reduced the

falling velocity by ~ 3% compared to the heated but uncoated copper sample.

When the surface temperature of the samples was increased to 180 °C, stabilization of the falling velocity of the Cu sample was observed: in the first section, the velocity reached 1.870 m/s, increased to 2.073 m/s in the second section, and stabilized at 2.027 m/s in the third section. The textured Cu-FeCl₃ sample formed a short-lived (~1 s) vapor film and reached a velocity of 2.181 m/s. The transient vapor film reduced contact with the liquid but did not create additional resistance, resulting in an optimal hydrodynamic regime.

When heated to 180 °C, the Cu + ZnOPMHS sample fell at 1.907 m/s in the first section and stabilized at 1.932 m/s in the third section. Meanwhile, the Cu-FeCl₃ + ZnOPMHS sample exhibited the lowest velocity in the first section (1.803 m/s), as the formed vapor film persisted around the sample for a relatively long time (in stationary conditions >10 s, see Table 3), increasing resistance and reducing the falling velocity by ~8% compared to the textured surface without coating.

3.6.3. Medium-temperature regime (200–240 °C)

At 200 °C, the Cu sample exhibited similar falling velocities in the first and second sections as at lower temperatures (1.858 m/s and 2.069 m/s, respectively), but in the third section, the velocity decreased to 1.883 m/s, which is attributed to sample cooling and increased fluid viscosity around it. The Cu-FeCl₃ sample formed a vapor film lasting 1–2 s and showed a lower velocity in the first section (1.815 m/s); however, after the film detached, the velocity increased to 1.972 m/s in the second section and 2.129 m/s in the third section. The Cu + ZnOPMHS sample formed a relatively thick (~0.881 mm; Table 3) vapor film and fell more slowly in the first section (1.892 m/s), then, after film collapse, reached velocities of 2.051 m/s and 2.076 m/s. The textured and superhydrophobic-coated Cu-FeCl₃ + ZnOPMHS sample formed a long-lasting vapor film of ~ 0.775 mm thickness, resulting in the lowest velocity in the first section (1.789 m/s). This is attributed to the vapor film increasing the effective hydrodynamic diameter of the sample and generating additional buoyant force (the “parachute effect”), which reduces the falling velocity. After the film detaches, rapid acceleration is observed along with a transition in the hydrodynamic interaction regime toward reduced friction conditions.

When the surface temperature was increased to 220 °C, the Cu sample formed a vapor film of < 0.4 mm and fell at 1.899 m/s in the first section; however, after the film disappeared in the second section, it accelerated to 2.169 m/s, and in the third section the velocity decreased again to 1.998 m/s due to sample cooling and increased fluid viscosity near the surface. The Cu-FeCl₃ sample formed a short-lived vapor film and reached velocities of 2.194 m/s and 2.184 m/s in the second and third sections, respectively, indicating that partial detachment of the vapor film reduces hydrodynamic resistance and allows more efficient flow around the moving body. Meanwhile, the Cu + ZnOPMHS sample formed a thicker (~0.887 mm; Table 3) and more persistent film, resulting in a lower velocity of 1.907 m/s in the first section, but later accelerating to 2.212 m/s. The Cu-FeCl₃ + ZnOPMHS sample exhibited similar behavior (1.915 → 2.236 m/s), indicating a mixed hydrodynamic interaction regime initial reduction in falling velocity due to lift forces, followed by acceleration after film collapse while the sample is still not fully cooled.

As the temperature was further increased to 240 °C, the polished Cu sample formed a thin and short-lived vapor film and fell at a velocity of 1.97 m/s in the first section; however, in the third section, the velocity decreased to 1.814 m/s due to increased hydrodynamic resistance as the sample cooled. The chemically textured Cu-FeCl₃ sample formed a vapor layer (and moves with a velocity of 1.809 m/s in the first section), and after its collapse, the velocity increased to 2.034 m/s. The Cu + ZnOPMHS sample maintained a more stable hydrodynamic interaction regime (1.904 → 2.043 m/s). Meanwhile, the Cu-FeCl₃ + ZnOPMHS sample formed the thickest vapor film (~0.891 mm; see Table 3), resulting in a lower velocity in the first section, but reaching the maximum velocity

recorded in the entire study in the second section of the channel (2.471 m/s). This indicates that after the collapse of the vapor film, while the sample is still hot, fluid viscosity near the surface decreases and flow around the sample improves, leading to a sharp increase in falling velocity. In the third section, the decrease in velocity is associated with sample cooling and the resulting deterioration of fluid flow around the moving body.

3.6.4. High-temperature regime (260–280 °C)

When the surface temperature of the samples was increased to 260 °C, the velocity of the Cu sample increased steadily (2.004 → 2.071 m/s), while the Cu-FeCl₃ sample exhibited the lowest velocity in the first section (1.809 m/s) due to the formed vapor film and its associated lifting effect. The Cu + ZnOPMHS sample fell more slowly in the first section (1.92 m/s), but later accelerated to 2.123 m/s, and in the third section, the velocity decreased to 1.911 m/s. A similar trend was observed for the Cu-FeCl₃ + ZnOPMHS sample (1.887 → 2.069 m/s). This indicates that a long-lasting and thickening vapor film initially reduces the falling velocity due to lift forces, but as the film thins or collapses, the fall accelerates. In the third section, the decrease in velocity is associated with sample cooling, which increases fluid viscosity and worsens flow around the moving body.

At 280 °C, the Cu sample formed a thin vapor film and reached its maximum velocity in the second section (2.327 m/s), which then decreased to 2.045 m/s in the third section, again associated with decreasing surface temperature and deteriorating flow conditions. The Cu-FeCl₃ sample did not form a vapor film, and therefore its falling velocity increased steadily (1.794 → 2.28 m/s), indicating a stable interaction regime between the moving body and the fluid. Meanwhile, the Cu + ZnOPMHS and Cu-FeCl₃ + ZnOPMHS samples formed long-lasting vapor films of ~0.781–0.872 mm (see Table 3), resulting in falling velocities of 1.901–1.98 m/s in the first section; however, after film collapse, the velocity increased to 2.148–2.203 m/s. This confirms that at higher temperatures, the Leidenfrost regime becomes unstable, and the breakdown of the vapor film, as well as its interaction with fluid flow around the moving body, leads to complex variations in hydrodynamic resistance.

It was also noted that the combination of surface texturing and a superhydrophobic coating promote the formation of a thick vapor film and enables the highest falling velocity (marked in green) at lower Leidenfrost effect temperatures.

After conducting the experiments, it was observed that the unheated polished copper sample (Cu) exhibited the lowest falling velocity among the investigated surface conditions, as highlighted in Table 4. In general, surface modification by etching, application of a superhydrophobic coating, or a combination of both treatments resulted in higher falling

velocities compared with the unheated polished copper reference. However, the effect was not uniform at all temperatures, and the velocity response depended on the specific surface condition and temperature.

The formation of a vapor film reduces direct solid–liquid contact and the associated viscous shear. However, it also increases the effective displaced volume and buoyancy and may modify the pressure drag. Consequently, a thicker or longer-lasting vapor film does not necessarily result in a higher falling velocity or a lower net drag coefficient. The observed hydrodynamic response is governed by the balance among reduced interfacial friction, buoyancy, gravity, and the evolving geometry and stability of the transient vapor film. This explains why bare copper may exhibit falling velocities comparable to or even higher than those of treated specimens at certain temperatures.

3.7. Drag coefficient

In this work, the drag coefficient of free-falling sphere samples Cu, Cu-FeCl₃, Cu + ZnOPMHS and Cu-FeCl₃ + ZnOPMHS was calculated based on the experimental data. This coefficient is a fundamental parameter essential for fluid hydrodynamics that shows energy, economy, and emissions savings. The equipment provided in Fig. 5 was used to conduct experiments. In the furnace, heated samples were released to fall freely in a water-tube channel. For the experimental results, the Basset-Boussinesq-Oseen expression was used to describe the spherical sample move in unsteady and low values Reynolds viscous flow. Drag coefficient C_D was established by using experimental data of the sphere's falling velocity, its diameter, water and samples densities. It was calculated according to expression [31]:

$$C_D = \frac{4gd(\rho_s - \rho_f)}{3v^2\rho_f} \quad (3.1)$$

Here: g – acceleration of free fall m/s^2 ; d – diameter of sphere, m ; ρ_s – density of solid material kg/m^3 ; ρ_f – density of liquid, kg/m^3 ; v – velocity of falling sphere, m/s ;

The Leidenfrost effect and formed vapor impact for drag reduction is provided in Fig. 11. The drag coefficient dependence from Re number is provided in Fig. 11. Experiments were conducted for spheres having Reynolds numbers between 3.5×10^4 and 4.5×10^4 . The Cliff and Gauvin correlation [32] for spheres representing C_D dependency from Re is well-established. However, due to differing experiment boundary conditions and surface treatment methods for the sphere samples, the comparison was made between unheated and heated samples up to a specific temperature at which the formation of vapor layer (or LE) was observed. Such comparative analysis between cold samples treated as a baseline allows to evaluate drag coefficient savings for the specimens having the same surface. Samples initial heating temperatures were 160, 180, 200, 220, 240, 260 and 280 °C. Spheres were free-falling in room temperature water of 20 °C degrees. In Fig. 11 all provided experiments red dots represent Leidenfrost effect formation under provided boundary conditions, while blue dots indicate cold samples that were treated as a base line for drag reduction savings.

For pure, uncoated and unheated Cu samples, the calculated drag coefficient value was 0.57 for free-falling spheres in a water-tube channel. For the same heated Cu samples at temperatures of 220, 240, 260 and 280 °C that formed the vapor film layer around the specimen, the drag coefficient values were 0.46, 0.47, 0.44 and 0.43, respectively. The calculated results show that increased initial heating temperature enables a stable vapor layer, which leads to drag reduction savings for pure copper, comparing unheated with heated specimens to 19.30%, 17.57%, 22.81%, and 24.56%, respectively.

For the chemically etched, unheated cuprum samples Cu-FeCl₃ the value of drag coefficient was 0.54. The lowest initial surface heating temperature at which vapor layer formation was marked as 180 °C and the vapor layer continued to form up to 260 °C. The drag coefficient C_D

Table 4
Overall falling velocity m/s of different samples at various temperatures.

Temperature/ sample type	Cu	Cu-FeCl ₃	Cu + ZnOPMHS	Cu-FeCl ₃ + ZnOPMHS
Cold	1.851 ± 0.00	1.908 ± 0.00	1.969 ± 0.00	2.012 ± 0.02
160	2.057 ± 0.04	2.103 ± 0.02	1.99 ± 0.00	2.057 ± 0.03
180	2.057 ± 0.03	2.057 ± 0.00	2.057 ± 0.01	2.057 ± 0.00
200	2.08 ± 0.00	2.057 ± 0.01	2.057 ± 0.00	2.034 ± 0.03
220	2.08 ± 0.02	2.128 ± 0.02	2.08 ± 0.02	2.057 ± 0.00
240	2.057 ± 0.01	2.057 ± 0.00	2.057 ± 0.00	2.314 ± 0.08
260	2.128 ± 0.00	2.152 ± 0.00	2.057 ± 0.00	2.103 ± 0.08
280	2.152 ± 0.03	2.057 ± 0.00	2.152 ± 0.05	2.103 ± 0.03

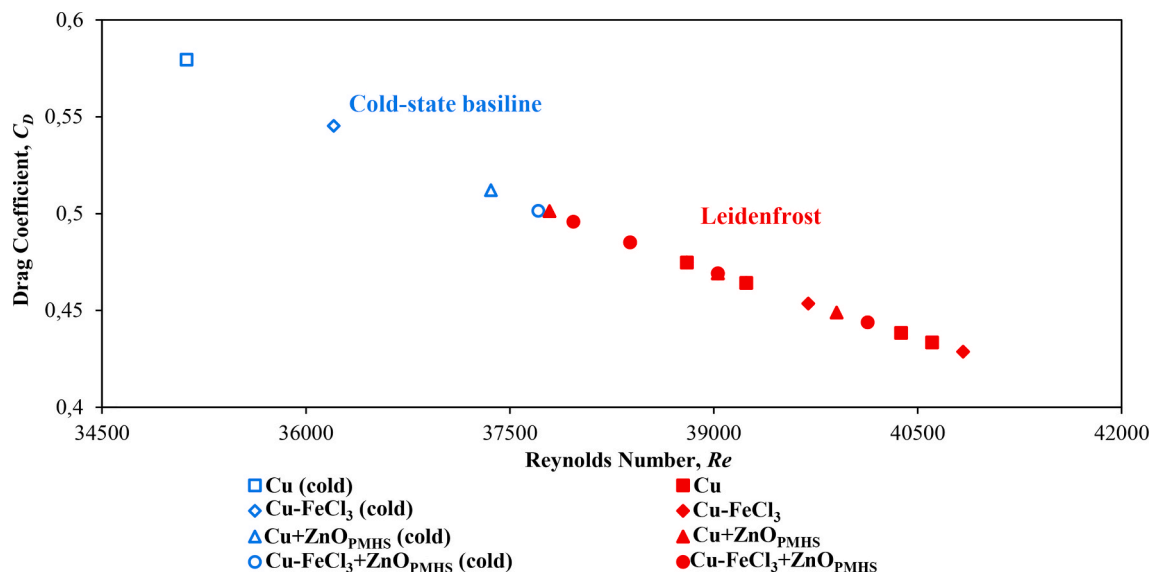


Fig. 11. C_D dependence from Re for cold and heated Cu, Cu-FeCl₃, Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples when initial heating temperature was 160, 180, 200, 220, 240, 260 and 280 °C, respectively.

values were changing in range from 0.46 to 0.42. This texturing effect leads to drag reduction comparing with unheated sample from 14.81% to 20.03%.

When experiments were conducted with cuprum samples coated with Cu + ZnO_{PMHS}, the lowest initial surface heating temperature at which the Leidenfrost effect took place was 160 °C. For coated Cu + ZnO_{PMHS} samples, a vapor film formed across the entire range of the investigated temperatures from 160 °C to 260 °C. In this case, drag coefficient C_D for unheated samples was 0.51. When experiments were performed with heated samples that formed a vapor layer around spheres, the drag coefficient was consistently decreasing in range from 0.50 up to 0.44. This allows to observe drag reduction in range from 2.34% up to 14.06%.

For experiments conducted with double surface treatment, applying both chemical etching with FeCl₃ and coating with ZnO_{PMHS} film, the calculated drag coefficient C_D for an unheated, free-falling sphere sample in a room-temperature water tank was 0.50. Such surface modification allowed to achieve vapor layer in the surface heating range from 160 °C to 240 °C. Two-stage surface treatment allowed to get a vapor layer lasting longer, therefore drag coefficient C_D was decreasing in range from 0.48 to 0.44. Compared with the unheated sample vapor layer allows to achieve drag reduction savings in range from 4% to 12%.

It is important to notice that the fastest specimen falling velocity and the thickest vapor layer does not guarantee the best drag savings. It is crucial to find the best boundary conditions for the optimal vapor layer formation and consider the ratio between the buoyancy and gravitation forces. This work main novelty was to identify how vapor film can form on differently treated and heated surfaces both in stationary setup (Fig. 4) and in a dynamic investigation stand (Fig. 9) when the specimen was falling in water-tube channels. Valuable insights of vapor layer formation open a way for future experiments when searching for the best optimal boundary conditions for vapor formation and drag reduction. A thicker vapor layer may seem advantageous, however, in some cases it should be evaluated with caution. For example, Cu in 240 °C forms vapor layer thickness of $\delta = 0.668 \pm 0.05$ mm. However, its falling velocity reached 2.057 ± 0.01 m/s, while the calculated drag coefficient was slightly smaller 0.47. In this case, a maximum falling velocity of 2.152 ± 0.03 m/s was noticed near 280 °C temperature, but the vapor film layer reached only $\delta = 0.342 \pm 0.005$ mm, which ensured drag coefficient of only 0.43. For Cu-FeCl₃, the maximum vapor layer $\delta = 0.757 \pm 0.05$ mm formed for the specimen when the surface was heated to 200 °C, sample velocity was 2.057 ± 0.01 m/s, and the drag

coefficient was 0.47. For the Cu + ZnO_{PMHS} case, the maximum vapor layer $\delta = 0.887 \pm 0.05$ mm formed when the temperature was 220 °C, however, the velocity reached only 2.08 ± 0.02 m/s. The performed experiment confirms that buoyancy forces overcome the gravity forces, therefore, a slight decrease of velocity could be noticed. Cuprum surface coated with Cu + ZnO_{PMHS} showed the highest velocity of 2.152 ± 0.05 m/s when the sample surface temperature was 280 °C. In this case, the formed film thickness reached $\delta = 0.872 \pm 0.05$ mm, and the drag coefficient value was only 0.44. For the textured and coated Cu-FeCl₃ + ZnO_{PMHS}, the maximum vapor layer was $\delta = 0.891 \pm 0.01$ mm when the surface temperature was 240 °C, and the specimen falling velocity was 2.314 ± 0.08 m/s. In this case, the drag value was only 0.39.

4. Conclusions

Experimental investigations into the formation of the Leidenfrost effect provided important data on the potential for vapor film formation and its influence on hydrodynamic properties at a water temperature of 20 °C. Measurements of the water droplet contact angle revealed that the best results were obtained with Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples, which exhibited exceptionally high superhydrophobicity ($>150^\circ$). Based on the analysis of SEM images, it can be stated that the tetrapod-like structure of the hydrophobic ZnO coating had a significant effect on vapor film formation around the heated surface. Therefore, the observed drag-reduction effects cannot be directly extrapolated to full-scale marine applications but demonstrate the potential of ZnO-based hydrophobic surfaces under Leidenfrost conditions. An important practical advantage of the ZnO_{PMHS} coating is its dual functionality: the coating can provide hydrophobicity and contribute to drag reduction under Leidenfrost conditions while simultaneously offering antifouling performance.

1. Analysis of cooling dynamics studies determined that copper samples coated with superhydrophobic ZnO + PMHS layers (Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS}) allow the formation of the Leidenfrost effect at a surface temperature as low as 160 °C, significantly lowering the critical temperature. Textured and coated samples, especially Cu-FeCl₃ + ZnO_{PMHS}, form a stable vapor film lasting over 10 s, with thicknesses of approximately 0.887 mm at 220 °C and 0.891 mm at 240 °C. The combination of surface texturing and superhydrophobic coatings effectively stabilises the vapor film, and

the enhancement in performance may be related to the morphology of ZnO tetrapods, which requires further investigation.

- It was established that the falling velocity of the tested specimens in the vertical water channel is determined by the interaction of surface tension, buoyancy, and fluid viscosity, as well as by the specimen temperature. In the first section of the channel, the falling velocity is the lowest because, once a vapor film forms, the buoyancy forces counteract the gravitational force and limit the falling velocity of the specimen. In the second section, upon detachment or breakdown of the vapor film and due to the reduced fluid viscosity, the highest falling velocity of 2.471 m/s is achieved, while as the specimen cools in the third section, its falling velocity decreases. The highest average falling velocity of 2.314 m/s was determined for the textured Cu-FeCl₃ + ZnO_{PMHS} specimen coated with a superhydrophobic coating and heated to 240 °C. This velocity was approximately 25% higher than that of the unheated, non-textured copper specimen (1.851 m/s). The obtained results confirm that surface texturing, the application of a superhydrophobic coating, and thermal treatment can significantly reduce hydrodynamic resistance and increase the falling velocity of the specimen.
- Experiments performed with different Cu, Cu-FeCl₃, Cu + ZnO_{PMHS}, and Cu-FeCl₃ + ZnO_{PMHS} samples revealed that the combination of chemically textured surfaces and superhydrophobic coatings can be used to reduce the drag coefficient. The study demonstrated that Cu + ZnO_{PMHS} and Cu-FeCl₃ + ZnO_{PMHS} samples were capable of reducing the Leidenfrost effect onset temperature from 200 to 220 °C to 160 °C, while simultaneously extending the vapor film lifetime to up to 3 s.
- The performed experiments have demonstrated that various copper (Cu) surface treatments, such as Cu-FeCl₃, Cu + ZnO_{PMHS}, Cu-FeCl₃ + ZnO_{PMHS} and different heating boundary conditions, have a significant impact on vapor layer formation around samples, leading to notable drag coefficient reductions. Overall, these results give critical insights about the relationship between vapor layer thickness, specimen velocity, and drag reduction and open the way for the application of this phenomenon in fluid dynamics.

CRediT authorship contribution statement

Lina Vorotinskienė: Writing – review & editing, Writing – original draft, Visualization, Supervision, Project administration, Investigation, Data curation. **Ainė Antanavičė:** Writing – review & editing, Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Data curation. **Monika Maziukiene:** Writing – review & editing, Writing – original draft, Resources, Investigation, Funding acquisition, Formal analysis, Data curation. **Raminta Skvorčinskienė:** Writing – review & editing, Writing – original draft, Supervision, Resources, Formal analysis. **Žygimantas Vainauskas:** Writing – review & editing, Writing – original draft, Software, Formal analysis. **Simas Račkauskas:** Writing – review & editing, Writing – original draft, Methodology. **Inna Pitak:** Writing – review & editing, Writing – original draft, Software, Methodology, Investigation. **Greta Srėbalienė:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Resources, Methodology, Investigation, Formal analysis. **Sebastiana Di Dio:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Monika Maziukiene reports financial support was provided by Research Council of Lithuania (LMT). If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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