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Advanced Methods for Investigating the Superheated State of Liquids Based on the Pump–Probe Principle

Artem N. Kotov*, Aleksandr A. Starostin and Aleksandr L. Gurashkin

Institute of Thermal Physics, Ural Branch, Russian Academy of Sciences, Yekaterinburg, Russia

*Corresponding Author: Artem N. Kotov. Email: artem625@mail.ru

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ABSTRACT: Dynamic methods for creating a superheated state of liquids and tracking their decay are presented. These methods allow relaxation characteristics of short-lived metastable states to be investigated across a wide range of temperatures and pressures. The relaxation of a medium is studied by a “probe” action after a short “pump” pulse. The concentration of the pump pulse in time and space allows the synchronization and localization of means for recording fast-flowing processes. Our aim was to carry out a brief review of methods for studying pulsed thermal processes in a superheated liquid based on the pump–probe principle by heating a platinum wire and releasing pressure in a bubble chamber. Although this article is a review in nature, we present new results obtained using the methods described. Non-stationary heat transfer to *n*-hexane with a water admixture was studied using the method of controlled two-pulse heating of a thin platinum wire. A water admixture of less than 0.01% increases the heat transfer of the probe by 10% at a sufficiently high degree of superheating of the base fluid. For uniformly superheated *n*-pentane, the dependencies of the refractive index and density on pressure and temperature were obtained using fiber optic densitometry. Using laser velocimetry, vaporization rates ranging from 4.8 to 9.5 m/s were obtained in the first microseconds of *n*-pentane boiling-up due to activation by a laser pulse in a bubble chamber at relatively low superheating (18–44 K).

KEYWORDS: Pump–probe; superheated liquid; activation boiling-up; *n*-pentane; laser pulse; fast-flowing processes

1 Introduction

In this article, we set out to describe the methods specifically developed for studying dynamic processes that accompany sudden changes in the thermodynamic parameters of the object under study. Such processes occur during phase transitions, as well as a result of shock thermal loads with large temperature gradients and pressure drops [1–6]. Such effects on liquid media are accompanied by phenomena associated with overheating of the liquid relative to the liquid–vapour equilibrium line $T_s(p)$ on the temperature (T)–pressure (p) phase diagram [1–3]. Conventional methods of studying the relaxation of the superheated state of a liquid are complicated by the random process of its boiling-up and large values of “lifetime” at low superheating $T-T_s$ [1].

As a rule, the development of dynamic processes is predicted using integrated models of physical systems. However, satisfactory results can only be obtained provided the presence of sufficient information about the thermophysical properties of objects comprising the system. When modeling non-stationary processes in relaxing media, account should be taken of changes in the properties of objects over time and temperature, which is a challenging task in the absence of reference data. The development of experimental methods makes it possible to obtain information about heat transfer directly from experiment in a timely

manner. For such measurements, experimental methods must be capable of localizing the application of thermal influence and measuring response signals.

An advanced approach to studying such phenomena in laboratory conditions involves the use of the pump–probe principle. In this approach, the pump pulse creates conditions for local thermal or mechanical disturbance in the sample, while the second probe pulse (referred to as a measuring pulse) allows the process of temperature relaxation to be monitored. The main feature of studying the response of the object under study to the induced pulsed local disturbance consists in the ability to synchronize the measuring system, which ensures the acquisition of data on the amplitude–time dependencies of properties with minimal dynamic distortions. The repeatability of the observed processes permits the accumulation of large arrays of experimental data while also making it possible to apply various techniques, e.g., stroboscopic imaging. Techniques for studying such processes rely on new capabilities of laboratory and technical facilities, which are based on modern achievements in electronics, lasers, and fiber optics [7].

In this work, we aim to carry out a brief review of the latest developments in the field of studying thermal processes on a small scale in terms of characteristic volumes and times (down to micro- and sub-microseconds) of superheated liquids using the pump–probe principle. In particular, we discuss the development of pulse methods for controlled short-term exposure of a microsystem with a superheated liquid and tracking its response at time intervals from 10^{-3} to 10^{-9} s. Methods involving pulse heating of a platinum wire and laser activation boiling-up through an optical fiber are currently being developed for investigating the behavior of superheated liquids. The method based on controlled heating of a platinum wire is efficient when studying the features of heat transfer in a probe-superheated liquid system using a two-pulse technique with adjustment of the heating electric current during the experiment. A modified method of bubble chamber facilitates the investigation of the fast-flowing processes of the initial stage of the phase transition of a superheated liquid, activated by a nanosecond laser pumping pulse.

Although this article is a review in nature, we present new results obtained using the methods described. Relative non-stationary heat transfer to *n*-hexane samples with water admixtures was studied near the boiling point using the method of controlled two-pulse heating of a thin platinum wire. For uniformly superheated *n*-pentane, the dependencies of the refractive index and density on pressure and temperature were obtained, which are necessary for estimating the rate of vapor phase formation upon activation boiling-up by a laser pulse. To the best of our knowledge, laser velocimetry was used for the first time to obtain the values of the phase transition rate in the first microseconds of *n*-pentane boiling-up upon activation by a laser pulse in a bubble chamber at relatively low superheating values of 18–44 K.

2 Methods for Heating a Platinum Wire Based on the Pump–Probe Principle

2.1 Temperature Plateau Method

A thin platinum wire, as a source of thermal action, is highly convenient to use in pulse experiments [1,8–13]. A thin platinum wire heater is a thermoelectric resistor; therefore, it can be easily integrated into electrical circuits of various designs. The authors in [14] presented a method of controlled heating of a thin platinum wire for studying the thermal stability of unstable liquids. This method involves reaching the required temperature (hereinafter referred to as the average temperature of the heater) with a voltage pulse rather rapidly followed by maintaining the temperature (for a certain period of time) with a second stabilizing pulse of the temperature plateau type. A short heating electric pulse (pump pulse) is formed by a discharge to the wire of a bulk capacitor of the required capacity. This pulse imparts the energy needed to reach the set temperature to both the wire and the thin wall layer of the liquid. The second probe pulse of a complex shape compensates for the drop in the heat flow from the wire to the liquid (as a result of its heating), thereby

maintaining the average temperature of the wire at the selected level. In order to maintain the temperature on the measuring pulse, it was necessary to solve the problem of compensating for the time-varying heat flux by controlling the power curve during the heating process. The authors [14] implemented two variants of such control, including a tracking system with an analog feedback loop and a digital system with electric current curve adjustment. The described method has been successfully applied to study the nature of boiling-up in polymer liquids, as well as to evaluate the contribution of sample thermal degradation phenomena to this process.

Despite its advantages for thermal measurements, the limitation of this method was the need for periodic adjustment of the device to objects with significantly different thermal properties. In the analog mode, adjustment of the control circuits was required. In the digital mode, it was necessary to periodically perform the procedure of determining and adjusting the heating power curve. Subsequently, a simplified version of the temperature plateau method, but retaining its main idea, was developed—a two-pulse heating of a probe with direct electric current.

2.2 Two-Pulse Heating Method

The idea of the method is to make the platinum wire heater reach the specified temperature rather quickly followed by its maintenance at approximately the same value using relatively simple technological means. The parameter being measured is the temperature dependence of the heater on the second pulse, which characterizes heat exchange [15]. Fig. 1a demonstrates the principle of measurements using the two-pulse method. The first direct electric current pulse I_1 (pump pulse) with a value of I_2 heats the probe up to a selected temperature T_1 , then the current is switched to a second measuring pulse (probe pulse) with a significantly lower current amplitude I_2 . In cases where the platinum wire temperature reaches the set value T_1 during the measuring pulse, the heating process is completed in less than the limit time t_3 .

Fig. 1b outlines three stages of the thermal process characteristic of heating the platinum wire to a temperature close to the line of achievable liquid superheating. After switching to the measuring pulse at time t_1 , the platinum wire cools down monotonically (area I) due to the relatively large heat flow into the cold liquid, which exceeds the heat output of the heater ($q_w < q_{liq}$). When the wall layer of the platinum wire warms up, the heat flux decreases, and the process enters stage II ($q_w > q_{liq}$). At the moment of transition I–II, the condition $q_w = q_{liq}$ is fulfilled. When boiling-up of the liquid occurs near the platinum wire in the measuring section, stage III of rapid cooling appears due to the additional heat outflow for phase transition. Then the process continues until the limit moment t_3 . The shape of the temperature curve at the probe pulse stage is characteristic of a rapidly proceeding thermal process in the superheated liquid until the onset of the convection process. The two-pulse method has proven convenient for studying the not fully stable states of a superheated liquid across a wide range of superheating temperatures (heating modes are shown in Fig. 1a). For a successful application of the two-pulse method, the heating conditions for the liquid under study should be selected correctly. The software configuration of the experiment parameters allows selection of the conditions described above to identify the characteristics of heat transfer near the values of achievable superheating. The uncertainty of the method is mainly determined by the errors of signal comparators at moments t_1 , t_2 , not exceeding 1% in modern electronics.

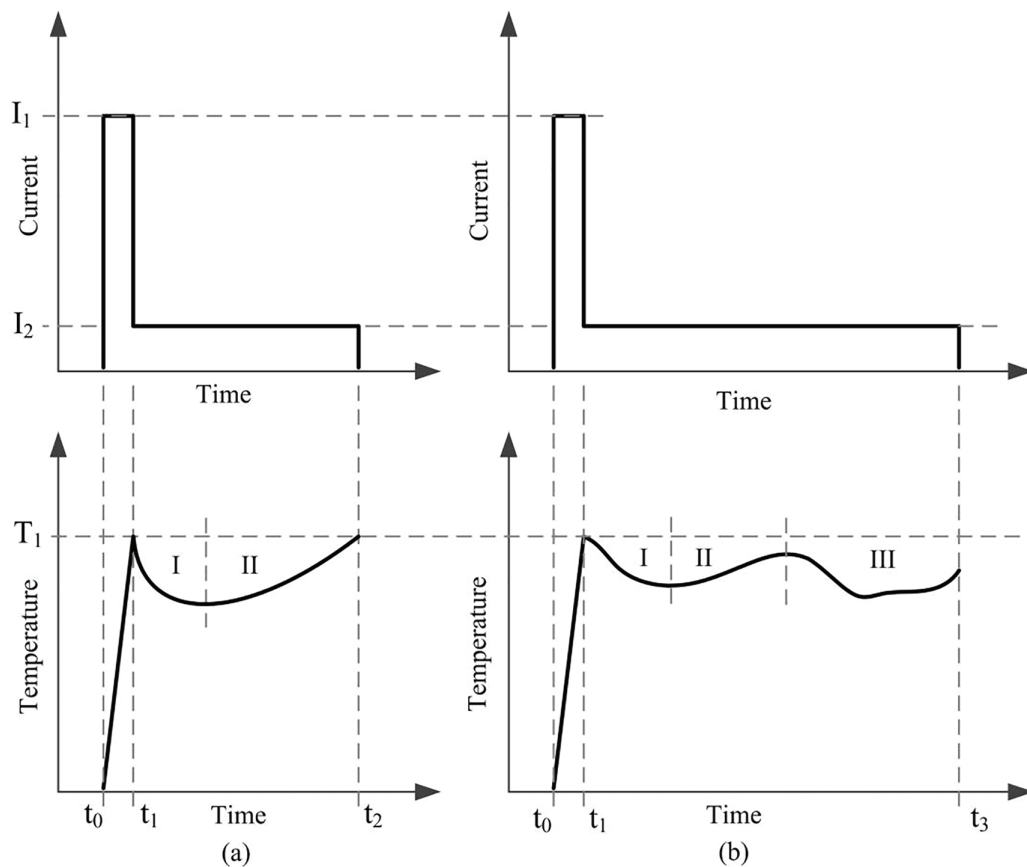


Figure 1: (a) The principle of two-pulse heating of the wire probe; (b) Heating process with spontaneously boiling-up of liquid sample. I—wire probe cooling at $q_w < q_{liq}$, II—wire probe heating at $q_w > q_{liq}$, III—wire probe cooling after liquid sample boiling-up.

2.3 Application of the Method of Two-Pulse Controlled Heating of a Platinum Wire to Study the Characteristics of Heat Transfer in Superheated Liquids

In [15], we presented a programmable device based on the two-pulse heating method for determining the relative heat transfer coefficient of a platinum wire in samples of liquid hydrocarbons with a low water content (Fig. 1a). The initial calibration was performed on dry samples with minimal water content. The measurement time for the second pulse was from 1 to 10 ms. The capabilities of the method were demonstrated using samples from a series of *n*-alkanes with the determination of the relative heat transfer coefficient of the platinum wire from dry and water-saturated samples with moisture concentrations ranging from 5 to 100 ppm by a compensation method. The compensation method [15] employs the possibility to achieve almost identical temperature–time conditions on the probe pulse for solutions with different concentrations of water admixtures during the experiment. In this case, the ratio of the heat transfer coefficients of the water-saturated and dry samples is virtually constant during heating on the second pulse, thus making this ratio a candidate characteristic of moisture concentration in the sample. A significantly disproportionate increase in the heat transfer of the platinum wire was observed at heating temperatures close to spontaneous boiling-up point of the liquid. The relative nature of the method allows minimizing uncertainties caused by uneven temperature distribution along the wire volume, the influence of external

fields, and wire geometry, provided that experiments are carried out on the same wire probe and at the same time.

For practical measurements, the device must ensure high repeatability of heating conditions, fine-tuning of the electric current amplitude of the second pulse and the heating temperature, as well as high-resolution recording of the temperature response. A repeatability of no more than 0.3%, adjustment with an uncertainty of less than 0.1%, and digital signal recording with an accuracy of 0.1% were achieved. The integrated uncertainty of the relative heat transfer coefficient after compensation was no more than 1%. Fig. 2a demonstrates the thermal effect of minor amounts of moisture (20 ppm) near the boiling-up point of a water-saturated sample and the principle of compensation to achieve the same temperature curve for samples with different concentrations. In the figure, curve 1 is a dry sample, current 0.46 A; curve 2—dry sample, current 0.43 A; curve 3—hydrated sample, current 0.46 A. To achieve a match between the temperature curves for 2 and 3, it was necessary to reduce the current for the dry sample by 30 mA. During the compensation procedure, in order to achieve the minimum root mean square deviation under the given experimental conditions, the step of change in the probing pulse current was 0.5 mA. The experimental results in Fig. 2b show an increase in the relative heat transfer coefficient up to 10% near the spontaneous boiling-up point with an increase in the moisture content of the initial sample by less than 0.01% for samples of *n*-hexane (blue), *n*-decane (red), and *n*-hexadecane (green) [15,16].

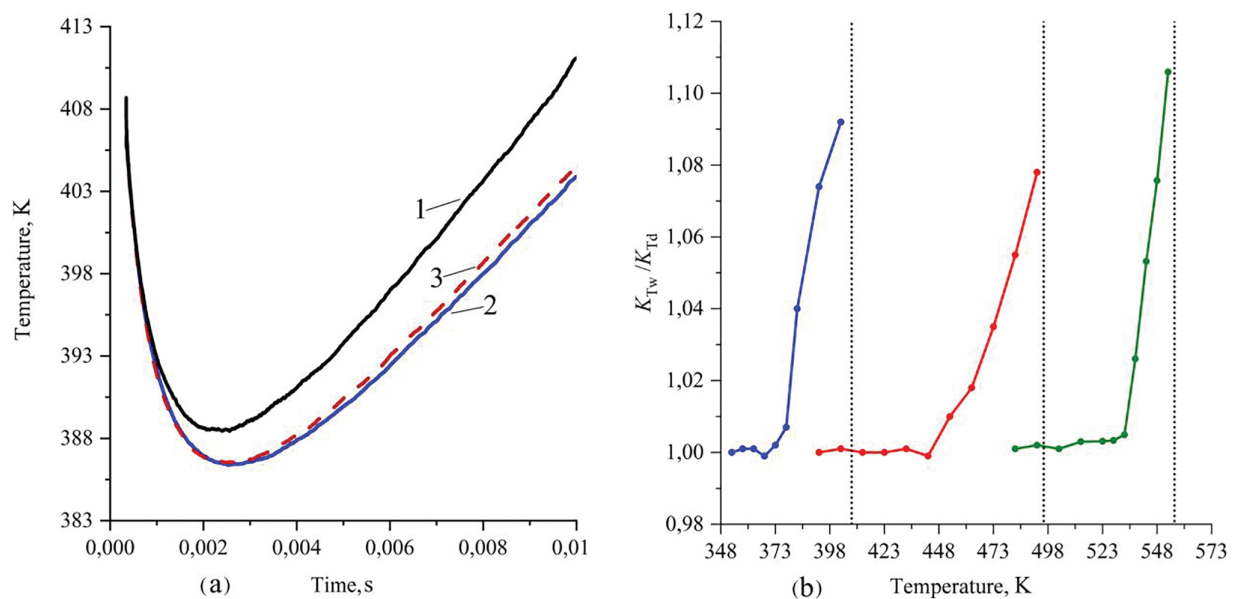


Figure 2: (a) Probe pulse heating thermograms with different currents I_2 for dry (1—0.46 A, 2—0.43 A) and water-saturated (3—0.46 A) *n*-hexane samples with compensation for temperature differences by reducing the heating current for the dry sample; (b) Dependence of the relative heat transfer coefficient of the wire probe on the temperature value T_1 for samples of *n*-hexane (blue), *n*-decane (red), *n*-hexadecane (green). The vertical lines show the corresponding boiling-up points of the water-saturated samples.

2.4 Advantages and Disadvantages of Wire Probe Methods for Studying Superheated States of Liquids.

Optical Probe Methods

A pulse-heated wire probe in a liquid forms a non-stationary temperature field around itself with a changing temperature gradient. The wire probe method has proven most successful in pulse measurements of the maximum overheating values of liquids on the surface of the wire. When the ratio of the wire length

to its diameter is greater than 50 (thin wire), it is possible to determine the temperature of the wire surface quite accurately (2%–3%) despite the natural distortions at its edges. However, in a liquid, the temperature varies greatly with time and distance. The dynamics of phase transitions can only be tracked by the integral change in the temperature (resistance) of the wire probe at least in the microsecond range due to the thermal inertia of even the thinnest wires. High-speed photography is limited by a time resolution of tens to hundreds of microseconds. At the same time, the classic ‘small bubble chamber’ method has long been used to study superheated liquids [1]. The initial state is achieved by stationary heating of the liquid under pressure exceeding the equilibrium pressure $p_s(T)$. The superheated state occurs after the pressure drop in the test volume. This yields a uniformly superheated liquid in a stationary metastable state with completely defined thermodynamic parameters. However, the liquid boils randomly, which makes tuning to the onset of the phase transition rather challenging. Our modification of the ‘small bubble chamber’ method involved addition of an SMF 9/125 single-mode quartz optical fiber as an optical probe. Owing to their high temporal resolution, spatial localization, and sensitivity, optical methods provide enhanced insight into the behavior of superheated liquids.

3 Optical Probing Methods

3.1 Fiber Optic Reflectometer–Density Meter (Densimeter) Method

The free end of a light guide immersed in a liquid can be used not only to deliver radiation into the medium, but also as a sensitive element of a high-speed optical sensor for monitoring the phase state of the medium in optical pump–probe methods. The effect of partial reflection of light from the light guide to the liquid interface can be used to obtain information about the properties of the medium in a thin boundary layer. The intensity of the reflected light is determined by Fresnel’s law as the difference in the refractive indices of the two adjacent media. The dependence of the refractive index of a liquid on its density makes the fiber optic reflectometer density sensor a convenient device for rapid determination of the current phase state of the medium.

The authors [17] applied the optical scheme of a fiber optic density meter to study evaporation and condensation, as well as transient processes in liquid carbon dioxide. In Fig. 3a,c, the reflectometer signal has a low level when the light guide is filled with a dense liquid phase. In Fig. 3b, the gas phase is characterized by an increase in the reflection signal, since the ratio of the refractive indices of the medium and the fiber increases. In Fig. 3d, in the transient mode during boiling-up, signal jumps are explained by the periodic capture of gas bubbles by the light guide. The fiber optic reflectometer has been successfully used to determine the density and to study spontaneous boiling-up in a uniformly superheated liquid in a clean bubble chamber [18].

Density is related to the refractive index of a liquid according to the Lorentz–Lorentz or Gladstone–Dahl formulas. To determine the refractive index, the voltage U was measured using a photosensor. The radiation reflected from the end of the optical fiber was directed to the photosensor, being related to the refractive indices of the quartz fiber n_{qz} and the liquid n_{liq} by the Fresnel ratio:

$$U = kI_r/I_0 = k[(n_{qz} - n_{liq})/(n_{qz} + n_{liq})]^2, \quad (1)$$

wherein k is the instrumental constant, I_r is the intensity of radiation reflected from the boundary, and I_0 is the intensity of radiation incident on the boundary. The constants and the refractive index of the light guide were determined in calibration experiments using reference data on the vapor–liquid saturation line. As a result, data were obtained for n -pentane and n -hexane on density across a wide range of superheated states

in terms of pressure and temperature (for *n*-pentane data are presented in Fig. 4). When using low-noise laser diodes and photodetectors, the sensitivity of the method can be better than 0.1% [18].

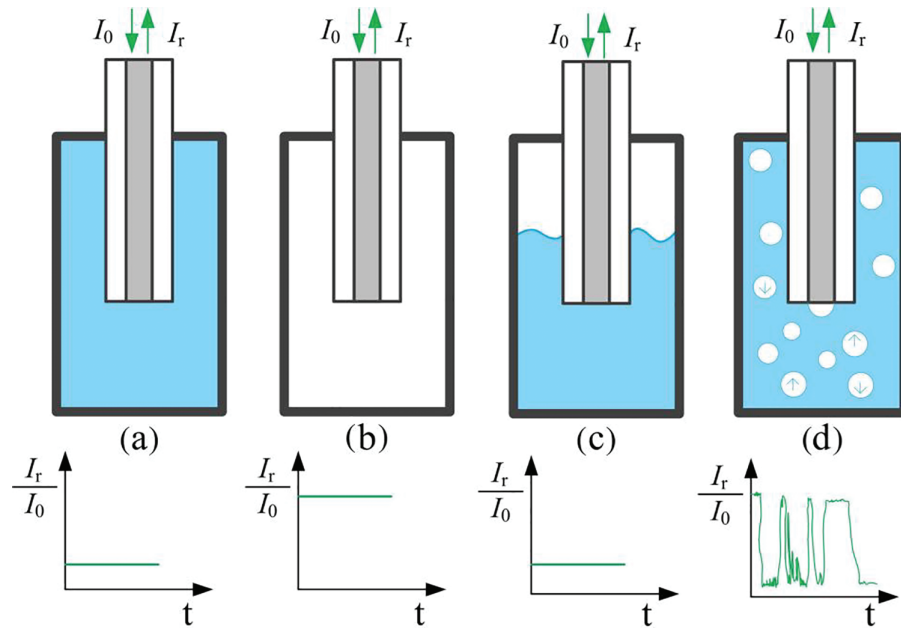


Figure 3: Demonstration of the operation principle of a fiber optic reflectometer for determining the phase state of a medium [17]. (a,c) The end of the fiber is in a liquid, the reflection signal is low; (b) The signal level is high, since the medium surrounding the end of the fiber is a gas; (d) The signal changes as the vapor bubbles reach the end of the fiber.

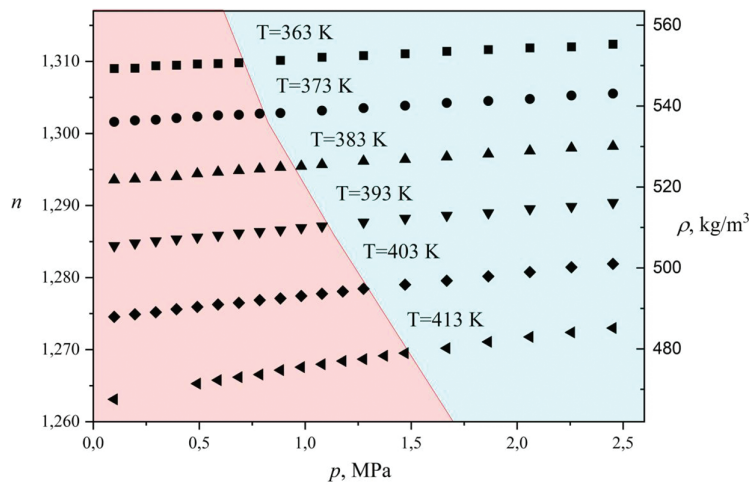


Figure 4: Experimental data for *n*-pentane: density and refractive index (at wavelength of 1.55 μm) at various temperatures and pressures. The red region corresponds to the superheated state of the liquid; the blue region corresponds to the liquid stable state.

3.2 Optical Boiling-Up Activation Method

Various factors, such as rapid local heating, local mechanical impact, ultrasound, or a powerful pulsed electromagnetic field, are capable of initiating the boiling-up of a superheated liquid (relative to the liquid–vapor equilibrium line) [19,20]. Concentrated pulsed laser radiation emitted from an optical fiber can create

conditions for the realization of all of the above factors [21]. In uniformly superheated *n*-pentane in a miniature bubble chamber, activated boiling-up is initiated by low-energy laser pump pulses, occurring at the end of a 10 μm diameter optical fiber [22]. The pump radiation has a wavelength of 1530 nm and a pulse duration of 1 to 100 ns. Activated boiling-up of *n*-pentane can be achieved across a wide range of superheated states, namely at a superheat degree of 15 to 105 K at atmospheric pressure. When the pump laser radiation has sufficient power, random boiling turns into initiated boiling-up.

Fig. 5 demonstrates the process of bubble growth at the end of the light guide after the action of a pumping pulse, as well as the process of reflectometer signal formation.

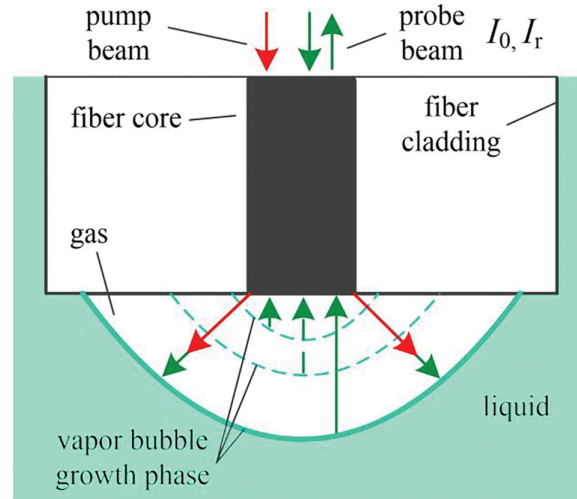


Figure 5: Growth of a vapor bubble at the end of a fiber optic cable after an activation pulse. The formation of reflected radiation from the reflectometer in the course of the growth of a vapor film is shown.

In the experimental setup, three different single-mode light guides were used inside the capillary of the bubble chamber. It was found that the light guides differ in the minimum pulse power that activates liquid boiling-up, although exhibiting similar threshold dependencies of lifetime in the superheated state on pulse power.

Activated boiling-up of *n*-pentane occurred at the end of the light guide simultaneously with the laser pulse and demonstrated a pronounced threshold character. When the superheat temperature was maintained, the threshold value was reproduced with an accuracy of 2%. However, the random nature of spontaneous boiling in response to the activating effect of laser pulses below the threshold value persisted. Even a series of pulses below the activation threshold did not affect the spontaneous boiling-up process, which reproduced the previous average “lifetime” values without activation.

3.3 Determination of the Speed of Transient Processes at the End of a Light Guide upon Activation Boiling-Up of a Superheated Liquid

During experiments on fiber optic densitometry of superheated liquids, we noticed that high-frequency oscillations of the reflected probe radiation occurred following the exposure to an activating pump pulse [22]. Since the frequency of the oscillations was greater than 10 MHz, the assumption of oscillations in the density of the medium was considered unlikely. A more likely explanation is related to the interference nature of the recorded periodic oscillations. Similar oscillations can be observed during the evaporation of a liquid film at the end of an optical fiber when reflections from the film and the end are superimposed. When the

activating pulse initiates a phase transition, a phase boundary forms and moves near the end of the light guide, reflecting a small portion of the probing radiation back into the light guide. Due to the difference in the refractive indices of the light guide material and the medium under measurement, the end face of the light guide forms another reflective boundary. The superposition of the two reflected waves results in a characteristic interferometer signal with a moving reflector (Fig. 6).

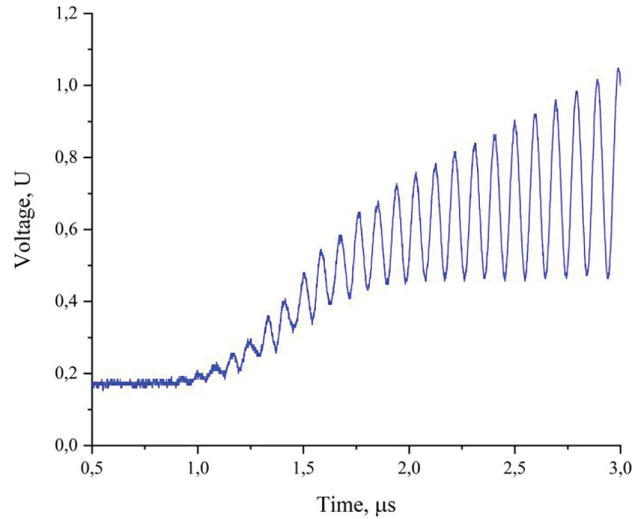


Figure 6: The voltage signal of a photosensor receiving the interference of reflected rays from the end of a light guide during the movement of the phase boundary after activation boiling-up of *n*-pentane at 338 K.

Doppler frequency F_D of the signal oscillations can be used to determine the movement speed of the phase boundary. In this signal, oscillations close to harmonics can be distinguished, with a frequency that depends on the speed of the reflector [22,23]. Thus, the movement speed of the phase boundary can be determined by the Doppler velocimetry ratio as follows:

$$V(t) = F_D(t)\lambda/2n(t), \quad (2)$$

wherein F_D is the current Doppler frequency shift (MHz), λ is the wavelength of the probing radiation (1.55 μm), $n(t)$ is the refractive index of the medium, which can be determined by the change in the average component of the signal received [18,23] taking into account the calibration of the fiber optic densimeter in accordance with Section 3.1 (Fig. 4).

The Doppler velocimetry method allows the growth rate of the vapor phase to be estimated in the activated boiling-up mode of *n*-pentane at various superheating temperatures [22]. The reflected signal of the probing radiation, measured by a photodetector, contains information about the change in the average refractive index over time, as well as the value of the Doppler frequency shift over time. Fig. 7 shows the frequency dependence of the phase boundary displacement rate on the activation boiling-up in *n*-pentane with varying degrees of superheating.

According to experimental data on Doppler frequency shift and refractive index measurements in superheated liquids, it is possible to determine the growth rate of the vapor phase taking into account the calibration of the fiber optic densimeter in accordance with Section 3.1 (Fig. 4). Initial velocities for different heating temperatures of superheated *n*-pentane were as follows: 327 K—4.8 m/s; 338 K—7.5 m/s, 353 K—9.5 m/s. Uncertainties in estimating the phase boundary velocity are mainly related to errors in determining $F_D(t)$ and $n(t)$ from experimental data. In the initial stage (Fig. 6), the fluctuations are insignificant compared

to the noise of the photodetector. This raises the velocity uncertainty up to 10%. A comparison of the bubble size (about 100 μm) using high-speed video and Doppler velocimetry data shows similar values with an uncertainty of 10%.

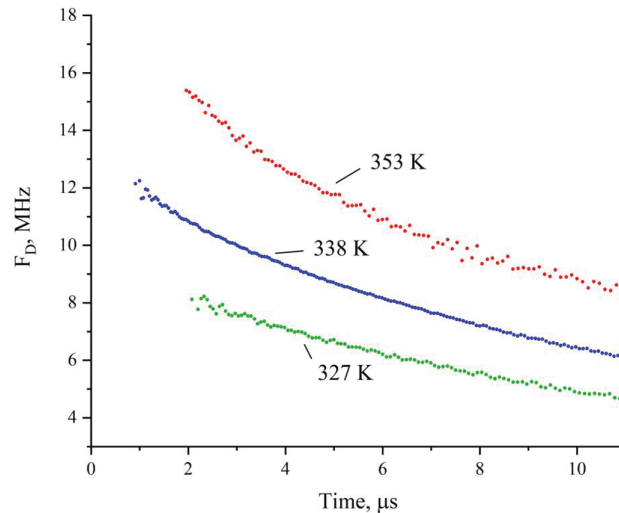


Figure 7: Changes in Doppler frequency over time during the propagation of a vapor front at different initial temperatures of superheated *n*-pentane. The countdown starts from the onset of the activation pulse.

4 Conclusion

It was already at the preliminary experiment stage that we noted the high potential of pump-probe methods for studying heat transfer in not fully stable states of liquids. The method of platinum wire is rooted in crossing the interphase equilibrium line by rapid heating during the first pulse. The subsequent probing pulse measures the thermal effects in the not fully stable region, where the “life” time of the fluid is limited. Two-pulse heating of a platinum wire with constant current is distinguished by its simplicity of implementation and stability of the heating mode. In the initial period of research into low concentrations of water in hydrocarbons, thermal effects were discovered that manifested themselves as a disproportionate increase in heat exchange during non-stationary heating of the wire probe. For a more detailed study, it was necessary to increase the sensitivity of measurements and ensure flexible control of the heating modes. The task of creating a programmable device that implements the method of two-pulse heating of a wire probe in a liquid with adjustable pulses was solved, which significantly expanded its scope of application. This device was used to derive a numerical criterion for the first time, namely the average relative heat transfer coefficient under similar temperature-time heating conditions, for comparing heat exchange in hydrocarbon liquids with small admixtures of water in superheated short-lived states.

Experiments on the activation boiling-up of superheated *n*-pentane were performed on a modernized miniature bubble chamber setup, which is typically used to study random boiling processes in uniformly superheated samples. The integration of a fiber optic system into the setup made it possible to study the phase transition with spatial localization at the micrometer level and temporal localization at the microsecond level. The transition from studying random boiling processes to that of induced processes made it possible to include the range of medium and low superheating degrees in the study. This regime is considered difficult to access due to the relatively long waiting time for the onset of a random boiling-up event.

New methods for investigating short-term states of liquids will enable more accurate, validated prediction of liquid behavior in processes involving high-power heat generation, where their thermal strength

plays an important role. Such processes occur, e.g., in both friction units employing liquid lubricants and electrical devices using insulating oils.

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Availability of Data and Materials: The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Ethics Approval: Not applicable.

Conflicts of Interest: The authors declare no conflicts of interest.

Nomenclature

F_D	Doppler frequency shift
I_1	Electric current value of the wire probe (pump pulse)
I_2	Electric current value of the wire probe (probe pulse)
I_r	Radiation intensity reflected from the fiber-liquid boundary
I_0	Radiation intensity incident on the fiber-liquid boundary
k	Instrumental constant of experimental setup
n_{qz}	Refractive index of the quartz fiber
n_{liq}	Refractive index of liquid under study
$n(t)$	Refractive index of the medium
T_1	Temperature setpoint of the wire probe under heat
$T_s(p)$	Liquid– vapour equilibrium line, where T —temperature; p —pressure
t_0	Startup time of the wire probe heat
t_1	End time of the wire probe first pulse
t_2	End time of the wire probe second pulse
t_3	Limit time of the wire probe second pulse
U	Voltage of the photosensor output
q_w	Power of the wire probe heating
q_{liq}	Heat flux into liquid liquid under study from the wire probe

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