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The Phenomenon of Superheat of Liquids: In Memory of Vladimir P. Skripov

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Abstract This article is devoted to the memory of Vladimir P. Skripov (1927–2006). He has received worldwide recognition for his monograph on metastable liquids published in 1972 (the English edition was published in 1974). We briefly discuss some studies deal with the phenomenon of attainable superheat of liquids and with measurements of thermophysical properties of liquids under conditions of a moderate degree of superheat. Main attention is paid to the studies performed by V.P. Skripov and his research group in the 1960s and 1970s. Experimental methods which provided breakthroughs in research on both spontaneous boiling-up kinetics and substance properties (the specific volume, isobaric heat capacity, ultrasound speed, and viscosity) in superheated states are presented.

Keywords Experimental methods · Superheat of liquids · Thermophysical properties

1 Introduction

A superheated liquid is a particular case of a metastable system supersaturated with respect to the phase equilibrium condition defined by the equality of chemical potentials of the phases at given values of temperature and pressure [1–6]. The phenomenon of superheat is observed when a system crosses the line of liquid–vapor equilibrium $T_s(p)$, where T_s is the saturation temperature and p is the pressure, without undergoing a phase transition. In this sense, a superheat initially precedes and then accompanies a boiling-up—the natural relaxation process in a superheated liquid. The degree of superheat determines the intensity of the process. The characteristic values of a spatial

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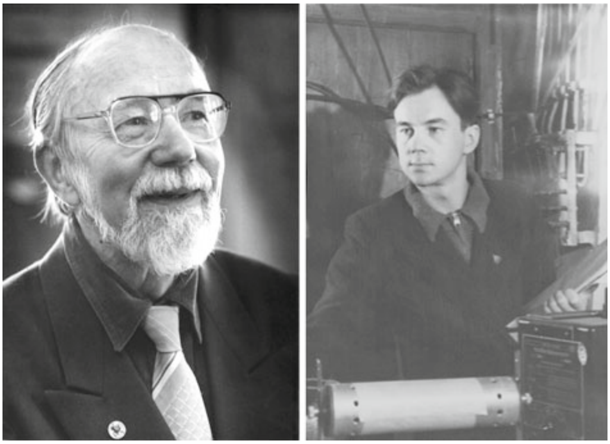

Fig. 1 Photographs of V.P. Skripov in 2005 and 1949

scale (r_c) and a temporal scale (τ), the so-called critical radius and lifetime of a system, respectively, serve as additional parameters of a thermodynamic description of superheated liquids.

The properties of fluids are usually investigated at stable states of a system. Such states are maintained as long as needed under constant external conditions and, therefore, are convenient for performing measurements. In spite of the fact that superheating is really "an everyday occurrence" [4], the region beyond the line of absolute stability of a fluid remains poorly known. The experimental difficulties are mainly caused by the presence of unidentified "weak spots" (nucleation centers) that trigger the boiling-up onset before the natural superheating threshold, predicted by classical nucleation theory [3,6], is attained.

This article is devoted to the memory of Vladimir P. Skripov (Fig. 1). He is the founder of the Ural thermophysical school in Ekaterinburg (formerly Sverdlovsk) [7–9], an informal community of researchers performing studies of metastable states of a different nature. Presently, this community carries V.P. Skripov's name. V.P. Skripov graduated from High School Number 10 in his native Velikiy Ustyug (a small town located in the northern part of Russia) in 1945. In the same year, he started his studies at the Physics Department of Moscow State University. Eight years later, on November 1, 1953, he came to Ekaterinburg for post-doctoral studies. At the Department of Physics and Technology, recently created at the Ural Polytechnical Institute, he initiated the investigations of the phase states with reduced stability using the methods of light scattering and boiling crisis at sufficiently high reduced pressures [3]. These investigations started a half-century travel into "the world of metastability."

In this article, attention will be paid to some studies dealing with the phenomenon of attainable superheat of ordinary liquids (results for cryogenic liquids, electrolytes, and polymeric liquids are presented elsewhere [10–14]) and with measurements of thermophysical properties of liquids under conditions of a moderate degree of superheat, when the lifetimes of a system are reasonably long. The discussion will be focused on the studies performed by V.P. Skripov and his research group in the 1960s to 1970s.

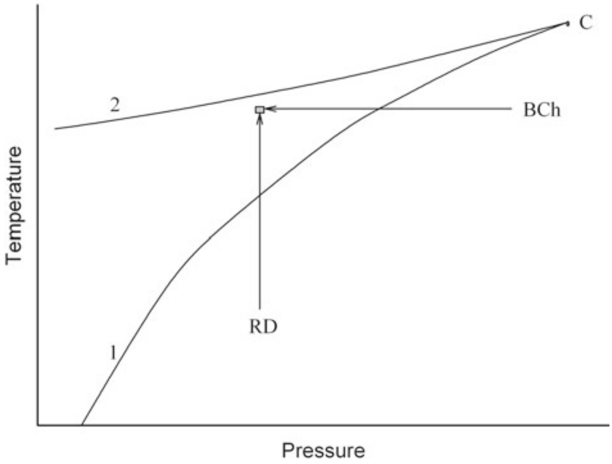

Fig. 2 Phase diagram for a liquid: (1) binodal $T_S(p)$ and (2) spinodal. C is the critical point. The trajectories entering the region of superheated states using techniques of a rising droplet (RD) and a small bubble chamber (BCh) are shown schematically

2 Attainable Superheat of Liquids

A superheated liquid has well-defined properties which can be measured experimentally. An understandable experimental limitation is imposed on the product of the volume of the superheated sample and the observation time, Vt_{exp}. In experiments, the superheated states can be reached using different methods. Two basic methods, presented with the techniques of a rising droplet and a small bubble chamber (see Fig. 2), are described below. These methods provided a breakthrough in the research of spontaneous boiling-up kinetics and the related phenomenon of attainable superheat in the 1960s [3,15]. The novelty of the approach taken was the recognition of the fact that the spontaneous boiling-up, a fluctuation-driven process, is a random event. Consequently, the experimental data (in the general case, the lifetime of the superheated state) should be averaged over a series of measurements under given external conditions.

In the first method, isobaric heating was achieved by means of having a sample liquid droplet rising in a host liquid, see Fig. 3. The sample and host liquids are mutually insoluble, and the host liquid has a substantially higher boiling temperature as compared to the one of the sample liquids. In most cases, sulfuric acid was used as a host liquid. A small droplet (less than 1 mm in diameter) floats up due to a preset temperature increase along the height of the tube filled with the host liquid. The droplet size is evaluated from the time period of its rise between two marks in the bottom section of the tube at a nearly constant temperature. At a certain moment of the rise, the temperature of the droplet exceeds the liquid–vapor equilibrium temperature for a chosen pressure value. At this moment, no reaction of the system is observed. In the course of a subsequent rise, the droplet explosively boils. In the experiment, the droplets boil in a narrow temperature interval, see Fig. 4, due to the strong temperature dependence of the nucleation rate

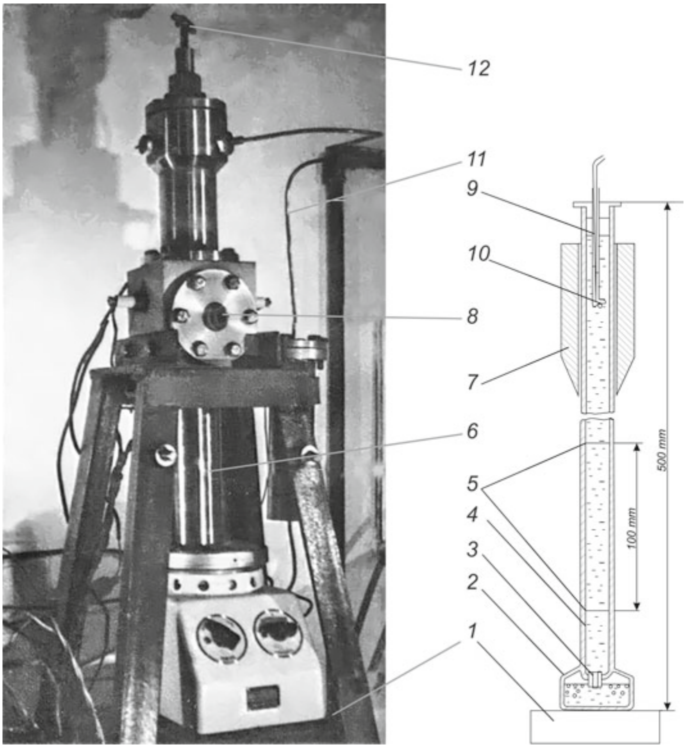

Fig. 3 Rising droplet technique. The stainless steel chamber for performing measurements under pressure (*left*), and the schematic of rising droplet device (*right*)—1: magnetic stirrer, 2: vessel with (sample liquid + host liquid) emulsion, 3: connecting capillary, 4: glass tube ($d_{\text{in}} = 15\,\text{mm}$), 5: marks, 6: stainless steel chamber, 7: heating block with two cutouts for droplet observation, 8: sealed glass window ($d = 15\,\text{mm}$), 9: mobile thermocouple envelope with glass "foot", 10: a droplet, 11: pressure line, 12: thermocouple driver

$$J\,(T,\,p) = (\tau V)^{-1}\,.$$

The classical nucleation theory predicts several orders of magnitude variation in $J(T,\,p)$ per 1 K near the superheating threshold [3,4,6,15]. As mentioned, the dependence of the lifetime of the superheated state on the temperature for a given pressure and droplet volume was determined. A mobile thermocouple envelope with a special glass "foot" for catching a droplet and keeping it under a chosen temperature ($T > T_{\text{s}}$) was used for this purpose. The mean lifetime τ_{m} was obtained by averaging over a series of experimental runs at each experimental point $(T,\,p)$. For a sufficiently small value of the $V\tau_{\text{m}}$ product $\left(<10^{-(2\pm1)}\,\text{mm}^3\cdot\text{s}\right)$, the measured attainable superheat values agreed with predictions of theory for the corresponding interval of $J(T,\,p)$ values. The concept of attainable superheat itself (as well as the term "superheat limit") is conditional in the sense that each mean lifetime corresponds to each value of temperature.

The device for studying the superheated liquids with this method built in the 1960s [16,17] is shown in Fig. 3. The device was used at pressures up to 30 bar, a sufficient range for the experiments with hydrocarbon liquids. This method has yielded interesting and unambiguous results on spontaneous boiling-up phenomena. However, it could not provide properties of a substance in superheated states other than the spontaneous boiling-up temperature.

In the second method, superheated states were reached via a nearly isothermal pressure decrease in a small $\left(V \sim 30\ \text{mm}^3 \text{ to } 50\ \text{mm}^3\right)$ bubble chamber designed for this purpose [3,15,18]. The chamber is a thick-walled glass capillary integrated with the electromagnetic pressure drop device and a thermostat. As in the previous case, the lifetime of the superheated liquid at different degrees of superheat was measured. The degree of superheat has been set by varying the experimental pressure $p_{\text{exp}} < p_{\text{s}}$, where $p_{\text{s}}(T)$ is the saturation pressure under a given temperature. The time counter is triggered by a pulse formed in the electromagnetic valve in the course of a pressure drop. The counting is stopped by the signal from a photodiode that monitors the boiling-up onset. Since the characteristic time of the phase transition is much shorter than the values of the lifetime observed experimentally, the time of the boiling-up onset closely approximates the lifetime. The modern version of this method is presented elsewhere [19].

The results on attainable superheat obtained with these two methods are in agreement for comparable $J(T, p)$ values, see Fig. 4. Complementing each other, these methods yielded a large body of data describing the spontaneous boiling-up temperature as a function of pressure, or, in more exact terms, the mean lifetime of the superheated liquids under well-defined (pVT) conditions. Figure 5 shows the results obtained with these methods for several substances. For generalization, the results are presented in reduced thermodynamic coordinates. The important point of these studies is the discovery of the approximate thermodynamic similarity of a large group of pure substances with respect to the spontaneous boiling-up conditions. In particular, the values of the reduced temperature, $T^*(p = 0.1\,\text{MPa})/T_{\text{c}}$, for the studied liquids lie within the narrow interval of 0.89 to 0.91.

3 Properties of Superheated Liquids

The second method described in the previous section has provided the means for maintaining a superheated sample (due to the replacement of the sulfuric acid environment by a more convenient glass), thus giving a basis for performing measurements of substance properties in superheated states, namely, the specific volume, isobaric heat capacity, ultrasound speed, and viscosity. Indeed, step by step, the suitable methods have been developed [3,15,17], as discussed below. Prior to performing measurements in superheated states, the corresponding devices were tested in experiments with reference liquids in the stable states.

For the *specific volume* (pvT data measurements) of a superheated liquid, according to the approach of Wismer [20], a glass piezometer was utilized with an original device for a rapid (quasiadiabatic) pressure drop similar to that of bubble chamber, see Fig. 6. The initial pressure $p_1 > p_{\text{s}}$ was set by the piston 1 when the magnet was switched on. Superheated states were achieved via switching-off the magnet 12. The experimental pressure $p_{\text{exp}} < p_{\text{s}}$ was set by the armature 13 and load 14 in advance, when the

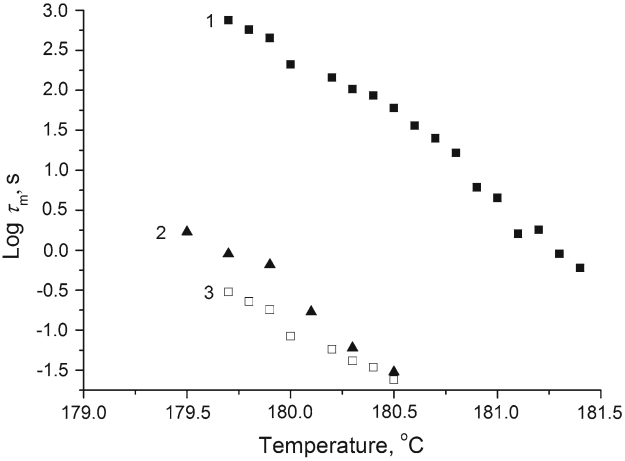

Fig. 4 Mean lifetime of superheated hexane in experiments with droplets, $V_1 = 1.2 \times 10^{-2}\,\text{mm}^3$ (points 1), and with bubble chamber, $V_2 = 30\,\text{mm}^3$ (2), at the pressure $p_{\text{exp}} = 0.1\,\text{MPa}$. For consistency of comparison, also shown (3) are the results of droplet experiments (1) recomputed to $V = V_2$ from the method of bubble chamber. $T_s(p_{\text{exp}}) = 68.7\,°\text{C}$

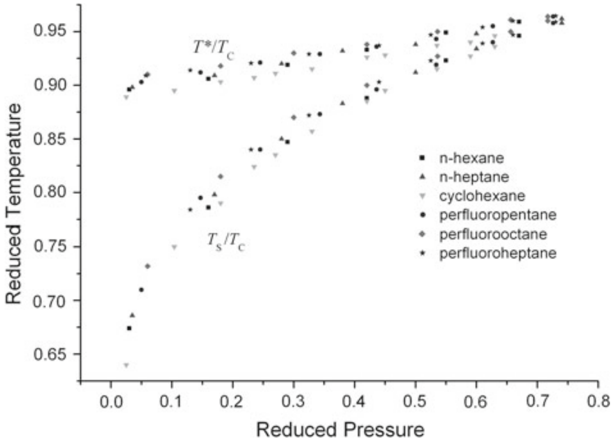

Fig. 5 Reduced spontaneous boiling-up temperature (T^*/T_c) obtained by the rising droplet technique and reduced vapor–liquid equilibrium temperature (T_s/T_c) plotted against reduced pressure (p/p_c), where T_c is the critical temperature, and p_c is the critical pressure, for several pure liquids—1: n-hexane, 2: n-heptane, 3: cyclohexane, 4: perfluoropentane, 5: perfluorooctane, 6: perfluoroheptane

magnet was off. A mercury seal was used for performing volumetric measurements. In general, other devices, such as a membrane, a sylphon bellow, or a piston, can be used [17]. The measurements may be carried out along isotherms, isochors, or isobars, according to the chosen type of device. In our case (see Fig. 6), the volume of liquid under investigation $\left(V \sim 10^2\ \text{mm}^3\right)$ is determined from the position of the mercury meniscus with respect to calibrating lines on the measuring capillary of the piezometer. Typical results obtained along the isotherms for both the stable and metastable

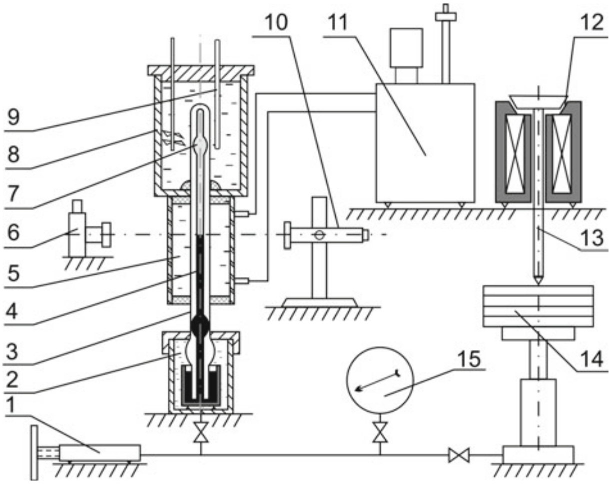

Fig. 6 Block diagram of the piezometer—1: piston device, 2: oil chamber for a pressure transfer, 3: measuring capillary, 4: mercury seal, 5: glass thermostat for the measuring unit of capillary, 6: photo camera, 7: sample liquid, 8: steel thermostat, 9: thermocouple envelope, 10: cathetometer, 11: temperature regulator, 12: electrical magnet, 13: magnet armature, 14: load, 15: manometer

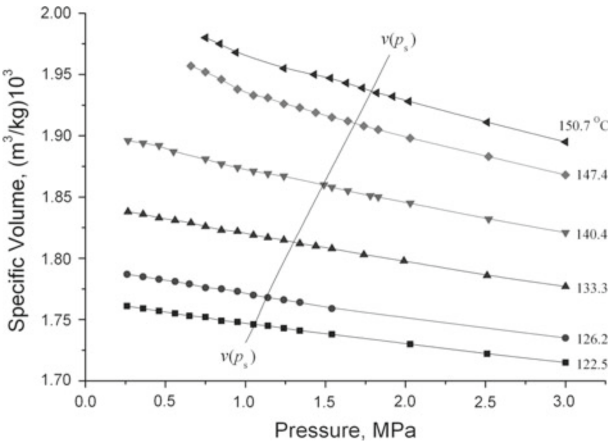

Fig. 7 Isotherms of the specific volume for diethyl ether in stable and superheated states at different temperatures. Subscript "s" refers to the values at the saturation line

regions are shown in Fig. 7. It has been found that the transition of a single-phase liquid across the line of liquid–vapor equilibrium is not accompanied by any peculiarities. The liquid as a whole does not "feel" the thermodynamically predicted equilibrium with the other macroscopic phase (at least, as long as this phase is absent).

On the basis of the results reported in [3,15–17], the following observations can be made. The isotherms, isochors, and isobars are continuous at the equilibrium curve; the isotherms and isochors exhibit nearly linear behavior up to the spontaneous boiling-up boundary. The isochors are preferable with respect to extrapolation of the experimental

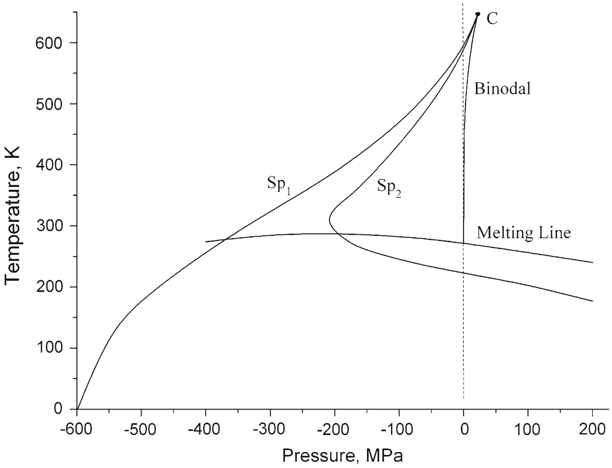

Fig. 8 Phase diagram of water. Two suggested shapes of spinodal (Sp$_1$ and Sp$_2$) are shown

data into the depth of the metastable region as thermodynamics does not impose any limitations on the isochors in this region. Therefore, the extent of their extrapolation may be larger than, for example, that of the isotherms.

Naturally, these results are valuable as they are. They certainly help to understand the behavior of liquids in the metastable region. Moreover, they have given key information for constructing empirical equations of state for metastable liquids (indeed, such equations have been constructed for a few liquids, including water [15,17,21,22]) and for approximation of such a fundamental element of a phase diagram as the spinodal. The latter has proven to be the envelope of a family of isochors in pressure-temperature variables. It should be added that, in the course of discussion of a possible shape of the water spinodal, V.P. Skripov substantiated the following point of view [23]: the spinodal is a monotonic curve with a positive derivative $(\mathrm{d}p/\mathrm{d}T)_{\mathrm{sp}}$ over the entire temperature range of its existence, see Fig. 8. The assumption that the spinodal of water has a hook-like shape (also shown in Fig. 8) prevailed at that time [6] and presently [24].

Ultrasound speed measurements were performed for a few liquids with the relative pulse method [15]. The superheated states were usually achieved via a pressure drop in the thermostatic acoustic cell. Ismagilov and Ermakov [17,25] performed ultrasound speed measurements for pentane, hexane, heptane, benzene, acetone, and diethyl ether. The glass acoustic cell with an inner diameter $d_{\mathrm{in}} \approx 10\,\mathrm{mm}$ and a length $l \approx 100\,\mathrm{mm}$ integrated with the hydraulic system was used. Similar to the specific volume data discussed above, the isotherms of ultrasound speed exhibit no anomalies at the liquid–vapor equilibrium curve, see Fig. 9. The values of the ultrasound speed and its first derivatives retain their continuity. Because the heat capacities are related to the speed of sound, one can expect that they would also retain the same features when crossing the liquid–vapor equilibrium curve. This point was confirmed experimentally, as discussed next.

Isobaric heat capacity measurements for superheated liquids were performed using an original flow calorimeter with a permanent flow direction [17,26], see Fig. 10. It

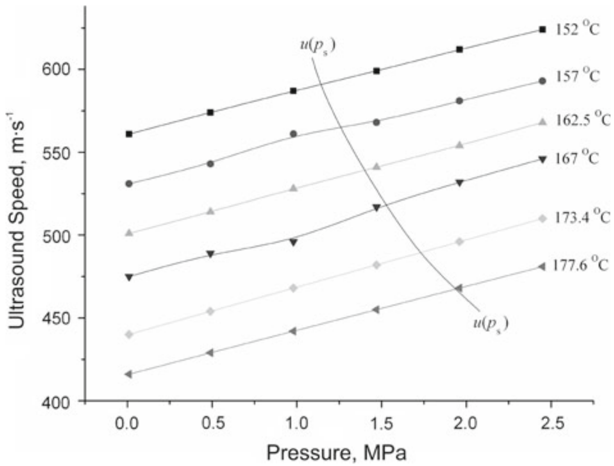

Fig. 9 Isotherms of the speed of ultrasound for acetone in stable and superheated states at different temperatures

consists of two small glass vessels $\left(V_1 \approx 2.8\,\text{cm}^3,\ V_2 \approx 1.0\,\text{cm}^3\right)$ connected by a measuring capillary ($d_{\text{in}} \approx 0.5\,\text{mm}$, $d_{\text{out}} \approx 1.5\,\text{mm}$, $l \approx 40\,\text{mm}$). The hydraulic system of the calorimeter includes the following elements forming a closed loop: a piston device for setting the pressure, a manometer, a plunger pump that maintains a constant flow rate m, and a flow switch providing one-way circulation in the setup. In the course of the experiment, the electric power W supplied to the measuring capillary (pos. 5 in Fig. 10) is set and the corresponding temperature increases $\Delta T = T_2 - T_1$ (pos. 9 on Fig. 10) of the sample liquid for given values of the flow rate, pressure p_{exp}, and temperature of the lower vessel T_1 are measured. The specific isobaric heat capacity c_p was obtained from the heat balance equation,

$$c_p = c_{p\text{s}} + (W - q_{\text{w}})\,(m\Delta T)^{-1}\,[1 - (\Delta T/\Delta T_{\text{s}})]\,.$$

Here subscript "*s*" refers to the values at the saturation line $T_{\text{s}}(p_{\text{exp}})$, where the heat capacity is assumed to be well known. Heat losses in the wiring q_{w} are determined experimentally at $\Delta T = 0$. Because the results are derived relative to the corresponding values in the saturated state, the terms associated with heat losses to the glass are cancelled. It was found that typical results for the heat capacity $c_p(p)$ of hexane at different temperatures are similar to those of $v(p)$. The dependence of the heat capacity on pressure has proven to be essential for isotherms at elevated temperatures, especially in the vicinity of the critical point. Most likely, the increase in c_p values with decreasing pressure is due to the proximity to the spinodal where the heat capacity becomes infinite.

In studies of *the viscosity* of a superheated liquid, we remember that the viscosity is the key property which determines the nature of the liquid state of a substance. A diagram of the simplest capillary viscometer providing penetration into the metastable region at ambient pressure is shown in Fig. 11 [15,27]. The liquid is superheated solely

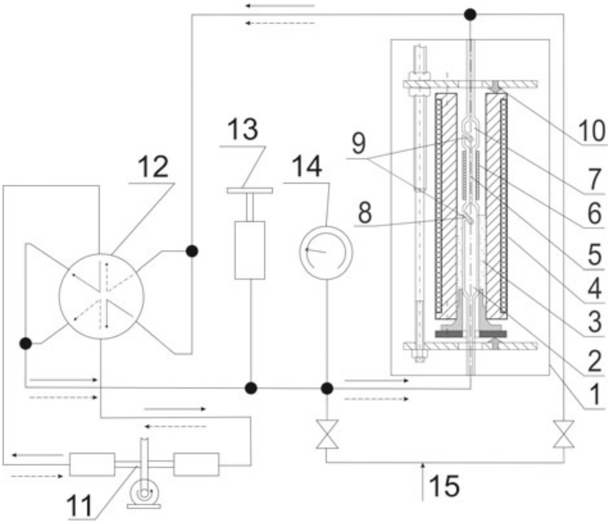

Fig. 10 Block diagram of the flow calorimeter—1: calorimeter, 2: lower vessel, 3: Ga–In alloy thermostat, 4: electric heater, 5: measuring capillary, 6: precise heater for the measuring capillary, 7: upper vessel, 8: thermocouple, 9: differential thermocouple, 10: vacuum chamber, 11: plunger pump, 12: flow switch device, 13: piston, 14: manometer, 15: line from filling device

in the capillary. The temperature in the vessels is maintained below the temperature of liquid–vapor equilibrium for the sample fluid. The sample fluid flows through the capillary (150 mm long and 0.24 mm in diameter) under a hydrostatic pressure. In the course of the experiment, the position of the meniscus is tracked, and the time period required for the meniscus to drop down by a selected value l is measured. Subsequently, the viscosity was obtained using the Poiseuille formula with appropriate corrections. The main experimental difficulty with these measurements is nonuniformity of the temperature along the capillary. Experimental errors associated with this problem generally increased with the degree of superheat (i.e., the temperature difference between the capillary and the vessels) and were difficult to control.

Typical results for the viscosity of liquids in stable and superheated states are shown in Fig. 12. As seen, the dependence of the viscosity on temperature demonstrates a smooth extension to the region of superheated states. The penetration into the region of superheated states is rather deep (a few tens of degrees). In order to elucidate the behavior of isotherms and isobars, measurements under pressure (up to 25 bar) were performed using a more complicated experimental setup [15,28]. It has been found that viscosity isotherms exhibit a linear pressure dependence. It was also suggested that the isotherms will retain such a behavior up to pressure values corresponding to the attainable superheat of the liquid.

4 Method of Pulse Heating

The previous section described the experimental methods and key results for studies of pure liquids in superheated states. However, in most practical applications, one

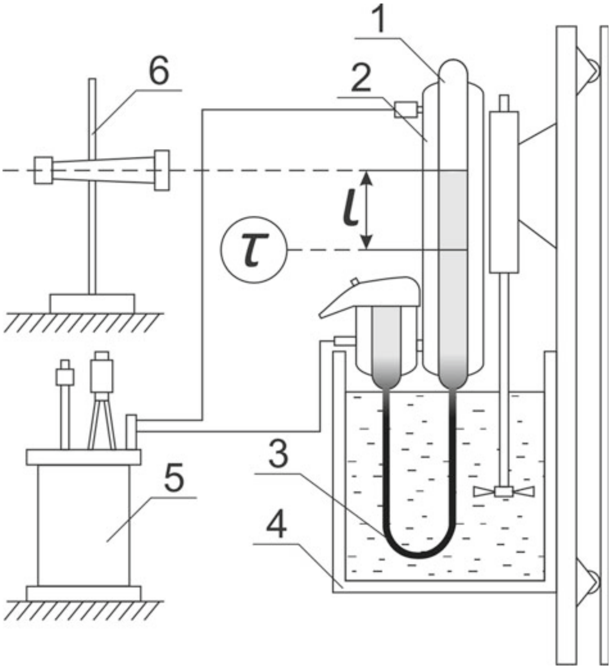

Fig. 11 Block diagram of the viscometer—1: vessels with the sample fluid, 2: glass thermostat ($T_1 < T_S$), 3: U-shaped capillary, 4: movable thermostat ($T_2 > T_S$), 5: temperature regulator, 6: cathetometer, τ: time counter

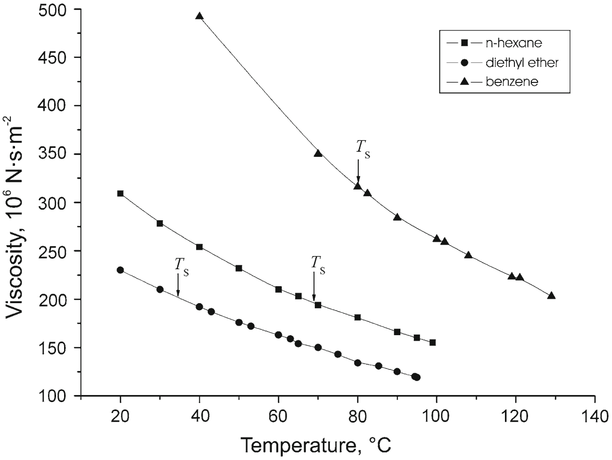

Fig. 12 Temperature dependence of the viscosity for several substances at atmospheric pressure. *Arrows* indicate the vapor–liquid equilibrium temperature (T_S) for each substance

deals primarily with mixtures. The addition of the second component considerably complicates the problem (and even more so for multicomponent systems). First, the liquid–vapor critical curve and, therefore, the saturation curves ($T_S(p, c = \text{const})$, where c is the concentration of the second component) shift toward higher pressures. Second, the thermodynamic compatibility of components may vary with temperature and pressure. These factors lead to a considerable extension of the two-phase equilibrium region for the mixture with respect to the pure liquid. Indeed, depending on the shape of the critical curve, the values of $T^*(p = 0.1\,\text{MPa})/T_\text{c}$ for mixtures vary over a much wider range, from 0.64 to 0.94, as compared with those (0.89 to 0.91) for pure liquids. Consequently, extended ranges of operation for an experimental apparatus developed for pure liquids is required. The latter point, in our opinion, is responsible for the surprising disparity between the size of the research field and the number of researchers working in this field.

Another example of the system associated with similar experimental difficulties is a liquid composed of long-chain molecules. As the chain length increases, the experimental measurements in superheated states become affected by thermal decomposition. As the size of the metastable region and the number of different relaxation times in a system increase, the use of pulse methods becomes more beneficial. The original pulse method for studies of the spontaneous boiling-up kinetics using a platinum wire probe with a diameter $d = 20\,\mu\text{m}$ and a length $l \sim 1 \times 10^{-2}\,\text{m}$ was developed in the 1960s [3,15,29]. The probe combines a heater and a resistance thermometer. This method is characterized by a short (from $10\,\mu\text{s}$ to 1 ms) heating time t and a small heated volume (due to the small wire diameter). Consequently, much higher values of the nucleation rate $J(T, p)$ are achieved as compared to those observed with quasi-static methods. High $J(T, p)$ values allow an unambiguous detection of the spontaneous boiling-up signal over the entire pressure range of the liquid–vapor coexistence region.

Discussed below is the modern version of this method which includes the devices for selection of the heating function and for recording of the corresponding response function over the heating range [30–32]. This approach provides fine control of the heating parameters, probe temperature $T(t)$, or power $W(t)$, taking into account the thermal properties of the superheated liquid. Correspondingly, the response function is the power $W(t)$ released in the probe for the given heating profile $T(t)$ or the resulting heating profile $T(t)$ for the given $W(t)$. The characteristic boiling-up signal on the heating curve (see Fig. 13) can be detected, and the heat-flux density through the substance $q = (W - W_\text{w})/(\pi dl)$, where subscript "w" denotes the part of power expended on heating of the wire probe, can be determined at any moment. The latter is the most important observable in pulse experiments with large changes in the probe temperature $\Delta T = T(t) - T(t = 0)$.

In general, the heating regime may be arbitrary. The parameters of the heating function are defined by the conditions of the problem. The most significant specific cases are the constant heating power mode ($W \approx$ const, see Figs. 13 and 14), the temperature plateau mode (thermostabilization of the pulse-heated probe at a given temperature value T_st), and the mode of microsecond thermal impact with subsequent probe cooling [30–32]. The first two modes allow direct comparison of the thermal resistance in the probe–sample systems, $R_\lambda(t) = \Delta T/q$, under identical heating conditions, namely, at a chosen temperature of a pulse-heated probe, or at a chosen heat release power. The third mode is used for the comparison of the short-time thermal stability of polymers and binders under conditions of shock heating.

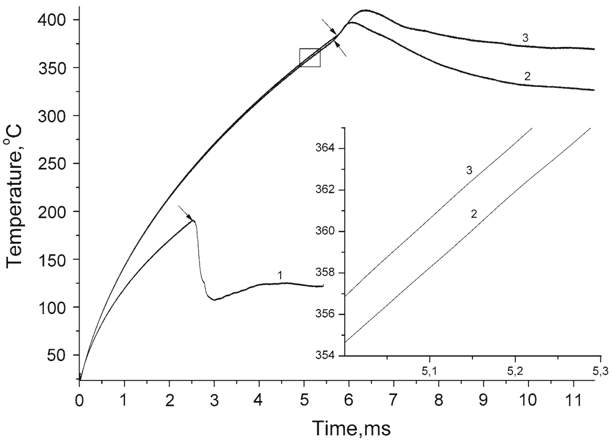

Fig. 13 Characteristic heating curves at constant heating power mode $W(t) = \text{const}$ for ethanol (curve 1, $W = W_1$), suspension of nanoparticles ($d \approx 10$ nm) of Al_2O_3 in the mineral compressor oil (2, $W = W_2$), and the oil (3, $W_3 = W_2$) at atmospheric pressure. *Arrows* indicate the timing of spontaneous boiling-up ($t = t^*$)

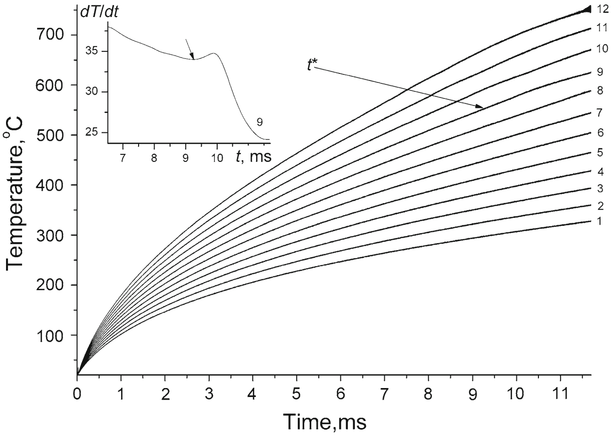

Fig. 14 Characteristic heating curves at constant heating power mode in polydimethyl siloxane PMS-350 with a gradual increase of power, from $W_1 = 1.69$ W (1) to $W_{12} = 3.25$ W (12), at atmospheric pressure. The arrow shows the timing of spontaneous boiling-up ($t^* = 9.245$ ms) for the representative case $W_9 = 2.83$ W (9). The insert illustrates identification of this moment following a characteristic perturbation (change in curvature) on the probe temperature curve

A turning point of the heating curve is the moment of boiling-up $t = t^*$, see Fig. 13. The section of the heating curve corresponding to the single-phase system ($t < t^*$) may be used for identification of the temperature-dependent thermophysical properties of the liquid from a single pulse experiment $\{P(t); T(t)\}$. One way of doing this is to apply a suitable evolutionary method based, for example, on a genetic algorithm [33]. The analysis of the "tail" portion of the boiling-up signal ($t > t^*$) is also used in a number of applications, such as the estimation of critical parameters for thermally unstable liquids [34] (including gas-saturated oils [35]), comparison of short-time thermal stabilities of polymeric liquids, and probing the content of volatile impurities in oil, including their traces [30–32]. The method allows performing measurements over wide range of conditions (pressure, temperature, composition of a mixture, regime of heating) and to study "inconvenient" substances, such as polymeric liquids, gas-saturated liquids, and nanofluids.

5 Conclusion

This article briefly describes only one of many areas of scientific research (e.g., see [5,8,9]) marked by pioneering studies of V.P. Skripov and his students. The results of his studies were published in four monographs and more than 300 journal publications. As expressed by Nakoryakov and Baidakov [9], due to the efforts of V.P. Skripov, his thermophysical school and his country became leaders in the area of metastable state research.

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