

Homogeneous Boundary of the Attainable Superheat of Liquids by the “Theory of Surges”

G. V. Ermakov,* M. A. Parshakova, and E. V. Lipnyagov

Institute of Thermal Physics, Ural branch of the Russian Academy of Science, Pervomaiskaya St. 91, Ekaterinburg, 620219, Russia

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An alternative theory of homogeneous nucleation based on the theorem on surges has been formulated. This theory gives a steeper temperature dependence of the homogeneous nucleation rate along isobars than that given by the classical Doring–Volmer–Frenkel–Zeldovich–Kagan theory. By the alternative theory, lines of attainable superheat lie much higher than classical ones and are close to the spinodal. Taking into account the dependence of the surface tension on r , i.e., the nucleus radius of curvature, assuming the power dependence of the Tolman length, $\delta = \text{const}/r^n$, from the theories for different liquids the boundary of attainable superheats moves toward lower temperatures.

1. Introduction

Ample experimental data have been accumulated with respect to the superheating of a great variety of liquids.¹ Trefethen,² Wakeshima and Takata,^{3,4} Moore,⁵ Skripov, Ermakov and Sinitsyn,^{6–12} Blander et al.,^{13–16} and Apfel^{17,18} used superheating of small droplets of a liquid in another liquid, which did not mix with the droplets and had a much higher boiling point. Wismer,¹⁹ Kenrick, Gilbert and Wismer,²⁰ Eberhart, Hathaway and Blander²¹ and Skripov, Sinitsyn and Baidakov^{22–24} superheated liquids in glass capillaries.

In works by Sinha and Jalaluddin^{25–27} and Skripov and Bulanov,^{28,29} liquids were superheated on glass or metal solid surfaces under quasistatic conditions. Pavlov and Skripov^{29–33} used pulsed superheating of liquids on thin platinum wires and brought the measured frequency of nucleus formation to 10^{24} – $10^{26} \text{ m}^{-3} \text{ s}^{-1}$. A present-day paper³⁴ studies the nucleation temperature at the surface of a thin metallic film immersed in water by the pulse method.

A method of measuring the boiling-up expectation time, which was developed by Skripov and Sinitsyn,^{35,36} has special significance among methods used to study superheating of liquids. This method allows determining and examining the random character of boiling-up of superheated liquids and also permits retrieving detailed information about the kinetic characteristics of liquids.

Experimental data, including those obtained at different pressures, are summarized in refs 1 and 37.

The basics of the classical theory of homogeneous nucleation were established by Gibbs. The main stades of development of this theory and the bibliography on this problem can be found in monograph.¹

The theory of homogeneous nucleation is based on two assumptions: boiling-up homogeneity, i.e., an equally probable formation of a steam nucleus at any point in a system, and the steady state of a random process leading to boiling-up. These assumptions were used to solve Zeldovich–Kagan’s equation

$$J = \dot{n}f(n) - D \frac{\partial f(n)}{\partial n} \quad (1)$$

where J is the nucleation frequency, that is, the number of viable

steam nuclei formed per unit volume in a unit time, n is the number of molecules in a nucleus, $f(n)$ is the distribution function, and D is the diffusion coefficient in the phase space. Solving eq 1 involves numerous approximations and simplifications. Specifically, the relation

$$f_n^0 = N_1 e^{W_n/kT} \quad (2)$$

(N_1 being the number of molecules per unit volume, W_n the work on formation of a nucleus with n molecules, k the Boltzmann constant, and T the absolute temperature) is taken for the equilibrium distribution function of nuclei with respect to the number of particles. As a result, the solution has the form

$$J = N_1 B \exp\left(-\frac{W_c}{kT}\right) \quad (3)$$

Here B is a kinetic coefficient characterizing the rate at which a nucleus overgrows the critical size, while the subscript c refers the quantity to a critical nucleus. The theories due to Doring, Volmer, Zeldovich–Kagan, and Deryagin–Tunitskii differ in the value of the coefficient B . E. N. Sinitsyn¹ analyzed those differences. He found that the coefficient B weakly depended on temperature and pressure in all approximations. The $N_1 B$ value could be assumed to be nearly constant, and a simplified formula could be proposed for practical calculations

$$\ln J = 88 - G \quad (4)$$

where $G = W_c/kT$ is the Gibbs number.

Results obtained by the aforementioned authors reduce mainly to the following.

Diversified liquids are easily superheated far above the boiling point. A maximum superheat temperature at atmospheric pressure is about $0.9T_c$ (T_c being the temperature at the thermodynamic critical point). Boiling-up of a strongly superheated liquid occurs randomly. If, for example, pressure and temperature are fixed, the time from establishment of an equilibrium state until boiling-up proves to be a random value governed by some law. Experimentally determined temperatures are in good agreement with the kinetic theory of homogeneous

nucleation. The discrepancy amounts just to fractions of a degree and does not exceed 3 °C for the superheat temperature of about 100 °C.

Comparing experimental and theoretical values, we can think of the theory at hand as rather accurate if it is assumed that homogeneous stationary boiling-up takes place in experiments. However, recent experimental facts cast some doubt on this statement.

In experiments concerned with the superheating of liquids, achievement of statistically reproducible values of the boiling-up expectation time was preceded by a technological "running-in" of the cell, which included nearly 100 or more preliminary boilings-up. During the "running-in" period, the mean boiling-up expectation time increased severalfold, while the superheat temperature reached some insurmountable threshold. Boiling-up of the liquid remained random, and the superheat temperature was relatively high throughout the "running-in" period, although boiling-up clearly occurred at weak points of the surface.^{1,38}

When metal, including porous, samples which enrich the system with surface boiling centers are placed in a glass cell, temperatures very far from and very close to the theoretical superheat temperature can be realized depending on the cell preparation method and the "running-in" time. In this case, the probability distribution density of the expectation time changes from an exponential dependence at the beginning of the "running-in" period to a dependence with a maximum at the end of the "running-in" period.^{38–40}

Theoretical estimates⁴¹ and direct visual observations⁴² show that superheat boundaries are determined by one weakest fluctuation center. Other boiling centers appear and increase in number with decreasing superheat temperature.

Studies of the activation kinetics of separate centers at low superheat temperatures corresponding to boiling-up of liquids yielded qualitatively similar results.^{43–45} The formation of bubbles at some boiling centers proves to be a random event. The probability distribution density of the boiling-up expectation time follows an exponential curve at a short activation time and a curve with a maximum at a long activation time. The effect of the boiling center "running-in" takes place. The temperature dependence of the mean activation time of a separate center is very strong and is comparable with a similar dependence at a high superheat temperature.

Homogeneous boiling-up of a liquid represents a limiting regime corresponding to the vanishing effect of such boiling-inducing factors as the vessel walls, ionizing radiation, ultrasound, gas dissolved in the liquid and adsorbed on the walls, etc. Therefore, one may think that the true boundary of homogeneous nucleation occurs at a temperature higher than temperatures observed in experiments. Proceeding from these considerations, it is possible to refine the existing theory and develop its nonclassical modifications. Some efforts have already been made in this direction.⁴⁶

2. Calculation of the Nucleation Frequency in Terms of the Theory of Surges

Let us consider density fluctuations in some fixed volume V of a superheated liquid. A surge beyond the steam phase density ρ'' means that a steam bubble of the given volume formed in the system. Assume that appearance of successive surges presents a stationary random process, while the fluctuation distribution density is normal. We shall use a ready result^{47,48} for this case and write the probability that no surge beyond the level ρ'' occurs in time t as

$$P_0(t) = \exp \left\{ -\frac{t}{2\pi} \left[\frac{\ddot{K}(\tau)}{K(\tau)} \right]^{1/2} e^{-(\rho'' - \bar{\rho})^2 / 2\sigma_\rho^2} \right\} \quad (5)$$

Here $\bar{\rho} = \rho'$ is the liquid-phase density, $K(\tau)$ is the time correlation function of the random process at hand, $\ddot{K}(\tau)$ is the second time derivative of the said function, $\tau = t_2 - t_1$ is the time interval in which a correlation is established, and

$$\sigma_\rho^2 = (\rho')^2 V^{-1} k T \beta_T \quad (6)$$

where k denotes the Boltzmann constant and T is the absolute temperature. Isothermal compressibility

$$\beta_T^{-1} = - \left(\frac{\partial P}{\partial v} \right)_T v'$$

can be found from a special equation of state describing a superheated liquid.¹

Assume that a system which deviates from the equilibrium position as a result of fluctuations always relaxes to the initial state following a usual exponential law in a characteristic time α^{-1} . In this case, it is also possible to use a ready result^{47,48} and write

$$K(\tau) = \delta_\rho^2 e^{-\alpha \tau} \quad (7)$$

The probability that a heterophase fluctuation appears in a time t is $P(t) = 1 - P_0(t)$. Hence, for the probability distribution density of the expectation time of this fluctuation, we have by definition

$$f(t) = \frac{dP(t)}{dt} = \lambda e^{-\lambda t} \quad (8)$$

where

$$\lambda = \frac{\alpha}{2\pi} e^{-(\rho'' - \rho')^2 / 2\sigma_\rho^2} \quad (9)$$

is the mean frequency of heterophase fluctuations in the volume V . The mean expectation time of these fluctuations follows from eq 8

$$\bar{\tau} = \lambda^{-1} \quad (10)$$

To calculate the homogeneous nucleation frequency J , assume that V is equal to the critical volume of a nucleus, V_c . In this case, the nucleation frequency of viable steam nuclei in the whole volume of the system, V_s , will be $\lambda V_s / V_c$. Referring this quantity to a unit volume and defining the nucleation frequency calculated in terms of the theory of surges as J_2 , we have

$$J_2 = \frac{\alpha}{2\pi V_c} e^{-(\rho'' - \rho')^2 / 2\sigma_\rho^2} \quad (11)$$

or

$$\ln J_2 = \ln \left(\frac{\alpha}{2\pi V_c} \right) - G_2 \quad (12)$$

where

$$G_2 = \frac{\left(1 - \frac{\rho''}{\rho'} \right)^2 V_c}{2\beta_T k T} \quad (13)$$

Let us use several methods to estimate the parameter α^{-1} . First, we shall take the molecular–kinetic relaxation time estimated by Nesis,⁴⁹ who considered flows of molecules condensing on the bubble surface and evaporating upon the bubble collapse. This estimate yields

$$\alpha^{-1} = R/\bar{v} \quad (14)$$

where R is the bubble radius and \bar{v} is the mean thermal velocity of molecules. It is readily seen that formula 14 coincides with a simplest and roughest estimate of the parameter α^{-1} . It represents just a relation between the characteristic length and velocity. At atmospheric pressure, this estimate gives, by the order of magnitude, $\alpha \cong 10^{11} \text{ s}^{-1}$ for the liquids considered below. To provide an alternative estimate, we assume that the density fluctuation vanishes thanks to self-diffusion of molecules. Then the change in the number of particles in a preset volume can be written as

$$\frac{dN}{dt} = -D \text{ grad } n \quad 4\pi R^2 \quad (15)$$

Here N is the number of molecules in a volume V , D is the self-diffusion coefficient in a liquid, R is the radius of the volume V , and n is the volume concentration of molecules. Substituting $N = nV$ and presenting $\text{grad } n$ as $(n - n')/\delta$, where the prime marks the volume density of the liquid phase and δ is the characteristic length, we obtain

$$\frac{d(n - n')}{dt} = -\frac{3D}{R} \frac{n - n'}{\delta} \quad (16)$$

Integration of this equation between some density n_0 , which corresponds to the initial moment of time, and n , gives

$$\frac{n - n'}{n_0 - n'} = \exp(-\alpha t) \quad (17)$$

where

$$\alpha = 3D/R\delta \quad (18)$$

Taking the self-diffusion coefficient $D \cong 1.4 \times 10^{-6} \text{ m}^2 \text{ s}^{-1}$ for similar liquids,⁵⁰ $R \cong 5.2 \times 10^{-9} \text{ m}^1$, and $\delta \cong 4 \times 10^{-10} \text{ m}$,⁵¹ we obtain $\alpha \cong 8 \times 10^{12} \text{ s}^{-1}$ at the temperature of achievable superheat at atmospheric pressure. If α changes by 3 or even 6 orders of magnitude, the superheat temperature is altered for fractions of a degree. In this connection, the parameter α need not be calculated exactly. The simplest formula (eq 14) will be used in further calculations.

3. The Comparison of the Classical Theory and the Theory of Surges

3.1. Basic Calculation Relations. The classical formula (eq 4) and an alternative expression (eq 12) will be compared for argon, *n*-hexane, and diethyl ether. We shall use known approximations for the work on formation of a critical nucleus and the difference of steam (P'') and liquid (P') pressures¹

$$W_c = \frac{16}{3} \pi \frac{\sigma^3}{(P'' - P')^2} \quad (19)$$

$$P'' - P' = (P_s - P') \left(1 - \frac{v'_s}{v''_s} \right) \quad (20)$$

In formulas 19 and 20, σ denotes the surface tension, P_s is the

saturation pressure, and v'_s and v''_s stand for the specific volume of the liquid and steam at their equilibrium line, respectively.

Expression 12 largely differs from the classical formula. The exponent does not contain isothermal compressibility β_T , which is directly connected with density fluctuations.⁵² An equation of state which would describe quantitatively the metastable region is required to calculate the above quantity.

3.2. Equation of State of Superheated Liquids. The problem of deriving an equation of state from first principles of statistical thermodynamics has not been solved even for stable liquids. Let us use equations of state based on thermodynamic relations and experimental P – v – T data for superheated liquids.¹

Integrating twice a known thermodynamic relation

$$\left(\frac{\partial^2 P}{\partial T^2} \right)_v = \frac{1}{T} \left(\frac{\partial C_v}{\partial V} \right)_T \quad (21)$$

from the saturation line (P_s, T_s) to some arbitrary state of a metastable liquid (P, T), we have

$$P(v, T) = P_1(v, T) + P_2(v, T) \quad (22)$$

$$P_1(v, T) = P_s(v) + \left(\frac{\partial P}{\partial T} \right)_{v/s} [T - T_s(v)] \quad (23)$$

$$P_2(v, T) = \int_{T_s}^T dT \int_{T_s}^T \frac{1}{T} \left(\frac{\partial C_v}{\partial v} \right)_T dT \quad (24)$$

Isochores of the liquid phase are nearly straight lines both in stable and metastable regions. Therefore, in eq 22, the linear temperature term (eq 23) is most significant, while eq 24 serves as a small correction.¹ The derivative $(\partial P / \partial T)_{v/s}$ in eq 23 is determined at the saturation line as a function of the specific volume using values of P , v , and T measured partially in the region of metastable states. Pressure and temperature at the saturation line, $P_s(v)$ and $T_s(v)$, are usually known from the literature and are interpolated with respect to the specific volume using some known or special method. To determine $P_2(v, T)$ explicitly, data on heat capacity C_v are also processed analytically, and the obtained dependence is used for calculating the right-hand member of eq 24 on the assumption that this dependence holds for the metastable region.

Calculations similar to those described above were performed for several liquids, and the corresponding equations of state can be found in a monograph.¹ The said group of liquids includes argon, *n*-hexane and diethyl ether, which are used here for comparison of the theories at hand. Equations of state for argon and *n*-hexane contain the correction $P_2(v, T)$. The equation of state for diethyl ether was derived only in the approximation of rectilinear isochores since the heat capacity C_v of diethyl ether is unknown. From the comparison of the equations of state and experimental data on P , v , and T , including those obtained in the metastable region, it is possible to determine the error of the specific volume. The error was 0.1% for argon at $105 \leq T \leq 140 \text{ K}$ and $0.1 \leq P \leq 5 \text{ MPa}$, 0.5% for *n*-hexane at $120 \leq T \leq 230 \text{ °C}$ and $0 \leq P \leq 3 \text{ MPa}$, and 1% for diethyl ether at $80 \leq T \leq 170 \text{ °C}$ and $0 \leq P \leq 4 \text{ MPa}$. So these equations of state are rather accurate and can be used for calculating isothermal compressibility and other thermodynamic properties of superheated liquids. In particular, they will be used below to determine the stability boundary for liquids (spinodal) from its definition $(\partial P / \partial V)_T = 0$. This is the best method for determining the spinodal, because it is based on experimental values of P , v , and T measured in the metastable region, which was found experimentally for the metastable section of recti-

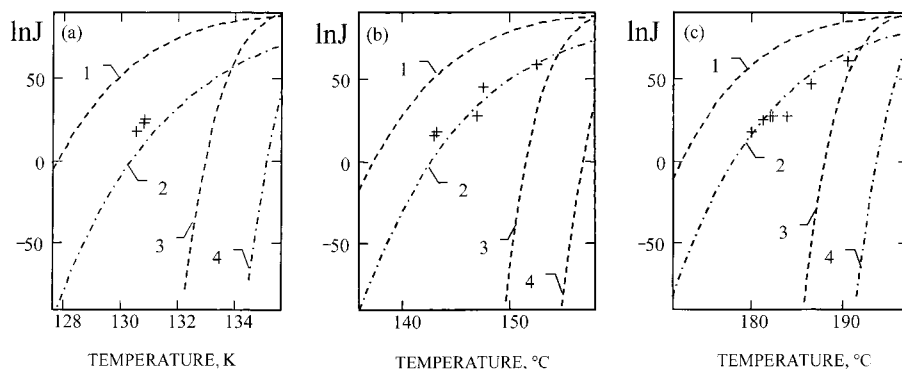


Figure 1. Temperature dependence of the logarithm of the nucleation frequency J ($\text{m}^{-3} \text{s}^{-1}$) at a pressure of 1 bar: (a) argon, (b) diethyl ether, and (c) n -hexane; (line 1) calculation by formulas 4, 19, and 20 of the classical theory taking into account the dependence of the surface tension on the nucleus surface curvature by formulas 27, 29, and 34 at $n = 0$; (line 2) as in line 1 but without taking into account the dependence of the surface tension on the nucleus radius; (line 3) calculation by formulas 12 and 13 of the theory of surges taking into account the dependence of the surface tension on the nucleus surface curvature by formulas 27, 29, and 34 at $n = 0$; (line 4) as in line 3 but without taking into account the dependence of the surface tension on the nucleus radius. \times denotes experimental points borrowed from (a) ref 23, (b) refs 3, 19, 20, 24, and 33, and (c) refs 3, 6, 8, 13, 22, 24, and 32.

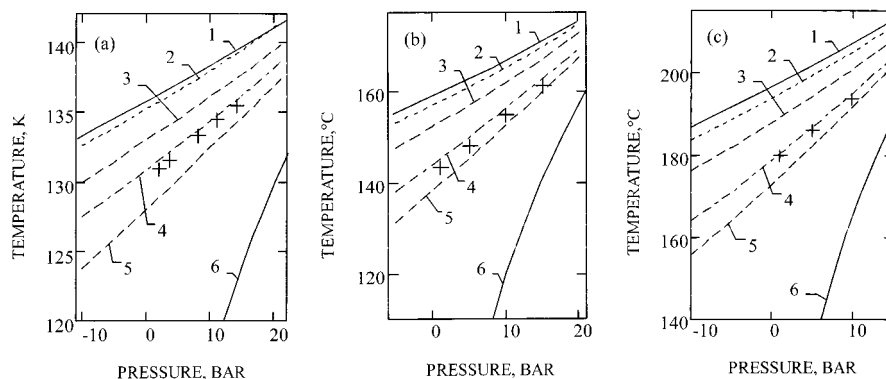


Figure 2. Achievable superheat boundaries at $J = 10^8 \text{ m}^{-3} \text{s}^{-1}$: (a) argon, (b) diethyl ether, and (c) n -hexane; (line 1) spinodal of liquids calculated from equations of state of the type 22–24; (line 2) boundary determined by formulas 12 and 13 of the theory of surges without taking into account the dependence of the surface tension on the nucleus radius; (line 3) as in line 2 but taking into account the dependence of the surface tension on the nucleus radius by formulas 27, 29, and 34 at $n = 0$; (line 4) boundary determined by formulas 4, 19, and 20 of the classical theory without taking into account the dependence of the surface tension on the nucleus radius; (line 5) as in line 4 but taking into account the dependence of the surface tension on the nucleus radius by formulas 27, 29, and 34 at $n = 0$; (line 6) saturation temperature vs pressure. \times denotes experimental points borrowed from (a) ref 23, (b) ref 24, and (c) ref 24.

linear isochores with due account of their small curvature, as seen from the data on isochoric heat capacity.

3.3. Results of Numerical Calculations and Their Discussion. Numerical calculations were performed using literature data^{53,54} on thermophysical properties of liquids. Cubic splines were used for interpolation of the properties with respect to temperature. Interpolation was accurate to within 0.5%.

Calculated dependences of J on temperature at a pressure of 1 bar are given in Figure 1. A much steeper dependence follows from the theory of surges. As a result, when the parameter α changes by 3 or even 6 orders of magnitude, the achievable superheat boundary, which is determined by the condition $J = 10^8 \text{ m}^{-3} \text{s}^{-1}$, shifts just for fractions of a degree. The difference in superheat temperatures of this boundary as determined in terms of the theories under discussion (curves 2 and in Figure 1) is considerable: 4.6 K for argon, 13.1 °C for diethyl ether, and 14.1 °C for n -hexane.

Figure 2 presents calculated boundaries of achievable superheat in T – P coordinates. The curve calculated by formulas 12 and 13 of the theory of surges is labeled 2, while the curve calculated using formulas 4, 19, and 20 of the classical theory is marked 4. From this figure it is seen that the boundary determined from the theory of surges is always above the classical boundary and is close to the spinodal labeled 1.

Gibbs defined the spinodal as a boundary where the surface tension and the tension surface radius turn to zero. Closeness of the achievable superheat boundary to the spinodal means that the dependence of the surface tension on the surface curvature should be taken into account in calculations.

3.4. Taking into Account the Dependence of the Surface Tension on the Nucleus Curvature Radius. This dependence can be found from the Gibbs–Tallmen–Kenn–Buff equation⁵¹

$$\left(\frac{\partial \ln \sigma}{\partial \ln r}\right)_T = \frac{2(\delta/r)[1 + (\delta/r) + (1/3)(\delta/r)^2]}{1 + 2(\delta/r)[1 + (\delta/r) + (1/3)(\delta/r)^2]} \quad (25)$$

Here $\delta = r_e - r$ is the Tallmen length, i.e., the distance between the equimolar surface r_e and the tension surface r . We then write the Tallmen length versus the tension surface radius as

$$\delta = b/r^n \quad (26)$$

where b is a constant. We introduce a new variable, $x = \delta/r = b/r^{n+1}$. Now eq 25 can be written as

$$\ln \frac{\sigma}{\sigma_\infty} = -\frac{2}{n+1} I \quad (27)$$

where σ_∞ is the surface tension at a plane interface

$$I = \int_0^x \frac{1+x+1/3x^2}{1+2x(1+x+1/3x^2)} dx \quad (28)$$

The integral (eq 28) can be easily calculated assuming $x > 0$. It takes the form

$$I = L \ln|x + A| + S \ln|B + Cx + Dx^2| + T \arctg(Ex + F) - W \quad (29)$$

The coefficients in eq 29 have the following values: $A = 1.7935$, $B = 0.55743$, $C = 0.80433$, $D = 0.66687$, $E = 1.45519$, $F = 0.87785$, $W = 0.64024$, $L = 0.22133$, $S = 0.139335$, and $T = 0.82228$.

To determine the constant b in formula 26, we consider the mechanical equilibrium condition in the limit $r \rightarrow 0$, which corresponds to closeness to the stability boundary of the liquid state

$$(P'' - P_{sp}') = \lim_{r \rightarrow 0} (2\sigma/r) \quad (30)$$

The limit in the right-hand side of eq 30 is calculated from formulas 27 and 29. The spinodal pressure P_{sp}' at a preset temperature can be found from the aforementioned equations of state for metastable liquids. Then the left-hand member of eq 30 can be calculated with good accuracy from eq 20. The pressure difference can else be found using an equation due to Fürth⁵⁵

$$P'' - P_{sp}' = 1.32\sigma_{\infty}^{3/2}(kT)^{-1/2} \quad (31)$$

This equation describes well the location of the spinodal in variables P and T . In this case, we have

$$b = 0.30429r_0^{n+1} \quad (32)$$

where

$$r_0 = \frac{2\sigma_{\infty}}{P'' - P_{sp}'} = 1.52 \left(\frac{kT}{\sigma_{\infty}} \right)^{1/2} \quad (33)$$

is the radius of a critical nucleus at the spinodal, which is found on the assumption $\sigma(r) = \sigma_{\infty} = \text{const}$. Thus, the Tallmen length is

$$\delta = 0.30429r_0(r_0/r)^n \quad (34)$$

Assigning different values to the exponent n in formula 27, it is possible to change the surface tension and the location of the superheat boundary. When $n \rightarrow \infty$ and $\sigma \rightarrow \sigma_{\infty} = \text{const}$, the superheat boundary is at its top position. When $n = 0$ and $\delta = \text{const}$, the superheat boundary occupies its bottom position.

When the dependence of the surface tension on the surface curvature is taken into account, the achievable superheat boundary calculated in terms of both theories shifts considerably toward lower temperatures.

Figure 1 presents calculated and experimental superheat temperatures for various nucleation frequencies. The dependence $\sigma(r)$ usually is disregarded in the classical theory and, therefore, one has to compare curves 2 and 3. When $J = 10^8 \text{ m}^{-3} \text{ s}^{-1}$, the superheat temperature difference of these curves is 2.6 K for argon, 8.5 °C for diethyl ether, and 8.8 °C for n -hexane.

Figure 2 depicts lines of the achievable superheat at $J = 10^8 \text{ m}^{-3} \text{ s}^{-1}$ and experimental data corresponding only to this frequency. As mentioned in the foregoing, the classical theory of homogeneous nucleation "takes advantage" over the experi-

ment if experimental results can be referred to homogeneous boiling-up of liquids. However, this assumption was not proved, but rather, some experimental facts point to the reverse.

It is highly probable that the effect of a hard wall limits the superheat temperature boundary of liquids. This is quite clear when liquids are heated in glass capillaries or on wires. The role of the surface is less clear when a liquid is superheated in another liquid. However, liquids interact chemically to a certain degree at high temperatures, and properties of the contact surface can change considerably. In some cases, the thermal effect is positive. For example, a hard carbon crust is formed on droplets of organic liquids when they are superheated in sulfuric acid. Moreover, dust particles, which act as a hard wall, are always present in the volume of a superheated liquid.

So the coincidence between results of the classical theory and experimental findings is significant only on the condition that a homogeneous character of liquid boiling-up has been proved. Such proofs have been lacking so far.

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