Pergamon

Int. J. Heat Mass Transfer. Vol. 37, No. 17, pp. 2771–2780, 1994

0017-9310/94 $7.00+0.00

0017–9310(93)E0124–Y

Boiling nucleation on a very small film heater subjected to extremely rapid heating

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(*Received* 24 *September* 1993 *and in final form* 15 *December* 1993)

Abstract—A very small film heater immersed in ethyl alcohol, toluene or water is heated at an extremely high rate to a maximum temperature rise of 93×10^6 K s^{-1}. The nucleation process is photographed and the temperature at boiling incipience is measured. At a higher heating rate, a large number of tiny bubbles generate concurrently on the heater surface and the temperature at boiling incipience agrees with the homogeneous nucleation temperature for ethyl alcohol and toluene, whereas it is about 19°C lower for water. The number density of the nucleated bubble is compared with those calculated by the nucleation theory.

1. INTRODUCTION

IT IS WELL known that a liquid must be superheated in order to boil. The degree of superheat is important both for industrial use and for academic interest: a great deal of research has been conducted which confirms that it is of an order of several or several tens of degrees for a practical application when a heating surface is used. Such a small degree is possible because of pre-existing gas entrapped in pits or in other sites on the heating surface acting as nuclei.

On the other hand, a large degree of superheating is required when no such pre-existing nuclei exist and explosive boiling occurs as a result of molecular density fluctuation. This type of boiling nucleation is directly connected to the limit of superheating of metastable liquid and is related to some industrial phenomena such as vapor explosion, flashing and so on [1, 2]. In this paper the term 'boiling nucleation' is used for nucleation by fluctuation and from a pre-existing gas [1].

Fluctuation nucleation is classified into homogeneous and heterogeneous nucleation and the process has been extensively investigated theoretically [3, 4]. A few methods are available to deal with it experimentally. The capillary tube method and the floating droplet method are substantially devoted to confirm the limit when a small number of nucleation occurs. The pulsed heating method, in which a heater immersed in liquid is heated very rapidly, may provide possibilities for approaching the whole state of the fluctuation nucleation phenomenon. Using this method, Skripov *et al.* [4–7] have carried out a series of experiments to demonstrate the nucleation behavior mainly in organic liquids, Derewnicki [8] in water, Sinha *et al.* [9] in liquefied gas, Okuyama and Iida [10] in liquefied nitrogen and Okuyama *et al.* in R-113 [11].

However, the rate of temperature rise in these experiments is limited and is less than about 11×10^6 K s^{-1} [6, 8]. At this rate of rise it is doubtful whether fluctuation nucleation occurs in water, where an extremely high rate of rise is needed, and it will be difficult to understand the dependency of nucleation behavior on the rate of rise. Furthermore, in almost all of these studies a fine wire has been used as the test specimen, with which making observations of the boiling behavior may not be adequate and the heat capacity of the wire greatly affects the temperature response when compared with using a film heater. Therefore, experiments with a flat film heater are considered to be promising for studying nucleation behavior, including aspects of boiling. However, only a few studies using a film heater have been performed so far. Asai *et al.* [12] have conducted experiments of rapidly heating a film heater with a guard film in order to apply the induced boiling phenomenon to a bubble-jet printer, but they have made no temperature measurement of the heater, which is indispensable for

NOMENCLATURE

J_{homo}	rate of formation of nuclei by homogeneous nucleation [1/(cm^3 s)]	$\bar{q}_{\mathrm{gen}}$	average heat production rate [MW m^{-2}]
J_{het}	rate of formation of nuclei by heterogeneous nucleation [1/(cm^2 s)]	$\bar{r}$	average rate of temperature rise [K s^{-1}]
		T	absolute temperature [K]
		t	time [s].
k	Boltzmann constant [J K^{-1}]		
N	number density of nucleated bubbles [m^{-2}]	Greek symbols	
		θ_{bi}	temperature at boiling incipience [°C]
N_0	number of molecules per unit volume [cm^{-3}]	θ_{l}	temperature of liquid [°C]
		θ_{w}	temperature of wall surface [°C]
n	bubble number in 0.1×0.25 mm^2 area	λ_{v}	heat of evaporation per molecule [J]
		λ_{l}	thermal conductivity of liquid [W (m K)$^{-1}$]
p	system pressure [Pa]		
p_{s}	vapor pressure at saturation temperature [Pa]	ρ	density [kg m^{-3}]
		σ	surface tension [N m^{-1}]
$\bar{q}_{\mathrm{l}}$	average heat flux to liquid [MW m^{-2}]	ϕ	contact angle.

this kind of academic study. Poppel [13] has tried to measure the temperature of a heater which is almost the same to that of Asai *et al.* but the measurement accuracy was not sufficient. The authors [14] have published a brief report on a peculiar type bubble generation which is observed when a rate of temperature rise higher than 10×10^6 K s^{-1} is applied to a film heater submerged in ethyl alcohol.

In this study, the authors have tried to heat a film heater immersed in a liquid with a much higher rate of temperature rise than those conducted up to now and to measure the temperature directly through detecting the electrical resistance. For this purpose, a very small film heater (0.1 mm wide × 0.25 mm long × 0.25 μm thick) was made and heated by direct current. The maximum rate of temperature rise attained in this study was 93×10^6 K s^{-1}, which is about ten times larger than that obtained in the preceding experiments using a fine wire. Observations of the aspect of boiling were also made through a microscope. Several facts were clarified regarding the behavior of boiling nucleation of ethyl alcohol, toluene and water under atmospheric pressure and some considerations were made by comparing the results with the theory of fluctuation nucleation.

2. EXPERIMENTAL APPARATUS AND PROCEDURE

In Fig. 1 a sketch of the test specimen is shown. The film heater is made by sputtering chromium and platinum of 0.05 and 0.20 μm, respectively, thickness on a quartz glass substrate. The width of the heater is 0.10 mm and the distance between two voltage taps is 0.25 mm. Since the width is 400 times the total thickness of 0.25 μm, the heater is regarded as a flat surface. Adoption of such a very small metallic film as a heater enables it to be heated with an extremely high rate of temperature rise and to measure the necessary quantities with high accuracy. Heat treatment was made to stabilize the temperature–resistance characteristics of the heater; it was once exposed to a 150°C de-oxidized atmosphere for 1 h and then to 500°C for 2 h. The surface of the heater was cleaned with acetone or trichloroethylene prior to the setup. A test liquid was distilled twice and boiled for 2 h to degas. It was poured into the space between the heater on the quartz glass facing upward and a cover glass set 5 mm apart from the heater. Experiments were carried out under atmospheric pressure with the liquid temperature maintained at 25°C.

The schematic diagram of the experimental apparatus is shown in Fig. 2. The heater is heated pulsewise by a pulse generator and amplified by a power amplifier. The voltage difference between the two voltage taps shown in Fig. 1 and the heating current are recorded by a storage oscilloscope via a differential amplifier and a current probe, respectively. The voltage and current are also transmitted from the oscilloscope to a personal computer. The average temperature of the film heater is obtained by referring to the measured electrical resistance and the calibration curve expressed by a polynomial equation. The calibration was carefully made by comparing the resist-

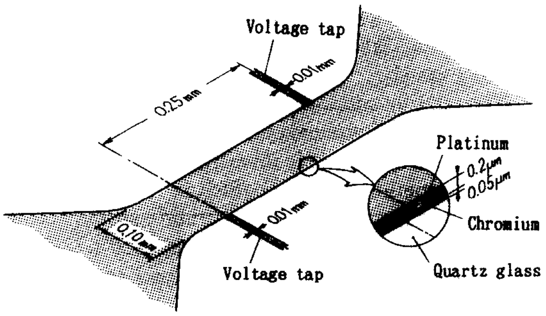


FIG. 1. Sketch of the test specimen.

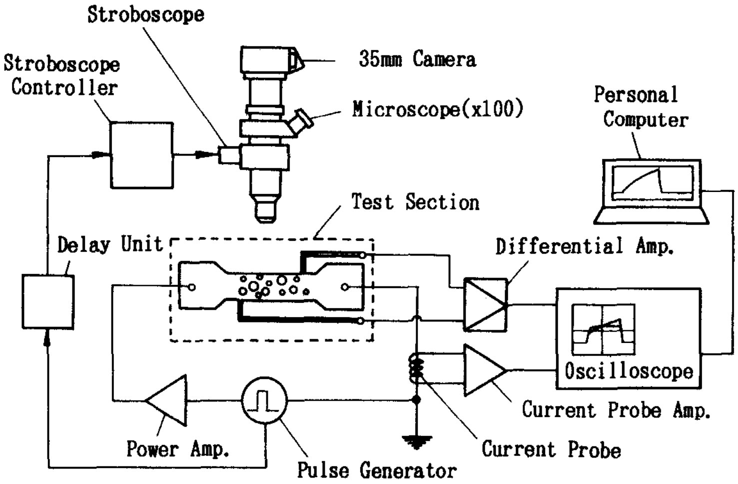


FIG. 2. Schematic diagram of the experimental apparatus.

ance with the indicated temperature of a standard platinum resistance thermometer securely attached to the specimen and heated in a thermostatic bath over the temperature range of interest. According to the simulation of one-dimensional heat conduction to the medium, the temperature difference across the film thickness is less than 1°C even at the maximum heating pulse in this experiment, and the measured average temperature can be regarded as the average wall temperature in this range. In addition, the wall temperature is regarded as the temperature of liquid in contact with the heater surface.

In Fig. 3, a typical example of the voltage difference and the current recorded by the oscilloscope for a single pulse shot is shown. Both quantities increase rapidly during the initial stage. After about 0.5 μs, the voltage difference increases but the current decreases gradually. Current is cut off at about 5 μs after the onset in this case, otherwise the heater would burn out. The pulse is supplied intermittently to the heater at a frequency of about 10 Hz.

With the extremely short and a very large power pulse, this experiment had a great difficulty in measuring necessary quantities. Great care was taken in setting the circuit constants, selecting and calibrating the probe and range, eliminating the noise, as well as other things. In addition, a series of calibrations and computer simulations to confirm the correctness of the measured values were made, and the error in the measurement of average heater temperature was estimated to be within ±5°C. This error is attributed to ±4°C due to the measurement error of the voltage difference and the current and ±1°C due to the error in selecting the exponent in the polynomial of the resistance–temperature calibration curve. Heat flux was calculated from the measured temperature and a non-linear one-dimensional heat conduction calculation, in which the power added to the heater was assumed to heat the heater itself and flow to both sides of liquid and quartz glass. Furthermore, each experiment was conducted by comparing the measured temperature change with the simulation result for the measured heat input. The aspect of boiling was observed through a microscope with 100 times magnification and photographed by a 35 mm camera with a 10 ns pulse stroboscopic light. The timing of light emission, i.e. the observation timing, was controlled by a delay unit which was triggered by the

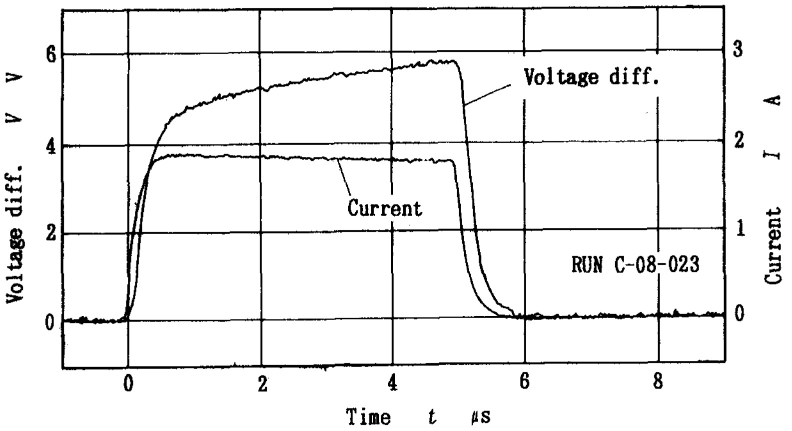


FIG. 3. Typical example of the voltage difference and current recorded by an oscilloscope.

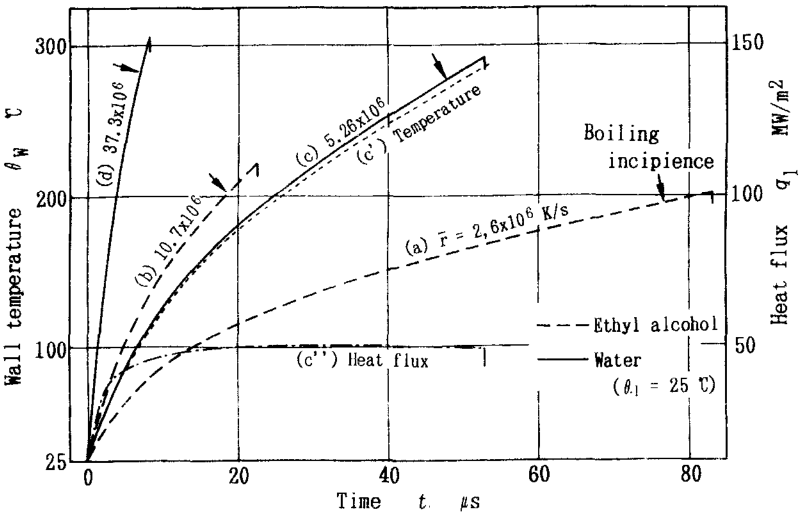


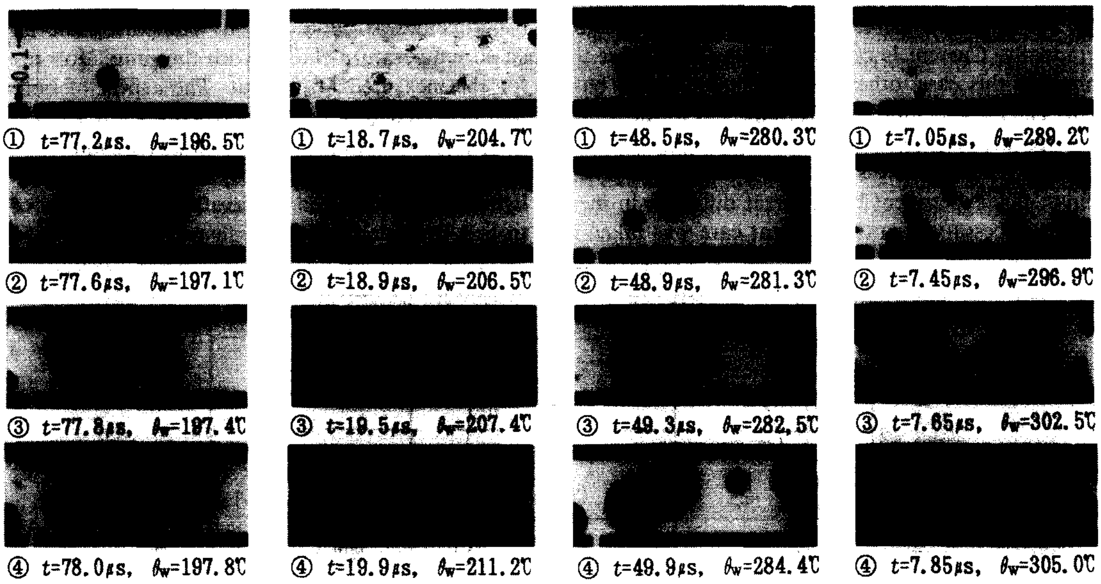
FIG. 4. Four typical examples of the wall temperature–time curve (they correspond to the four groups of photographic records shown in Fig. 5).

onset of an input pulse. An ISO3200 film was used and, in addition, an augmentative development equivalent to ISO20 000 was given to it.

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

3.1. *Temperature–time curve and aspect of boiling just after boiling incipience*

In Fig. 4, typical examples of the measured response of the wall temperature are shown. The four curves (a)–(d) in the figure correspond to the four groups of photograph shown in Fig. 5. Arrows indicate the conditions of boiling incipience, i.e. the instance when a bubble generation was recognized on the heater for the first time. One bubble on the heater corresponds to the number density of 4×10^7 m^{-2}. The average rate of temperature rise is defined as the ratio of the temperature difference between the temperature at boiling incipience and that of liquid to the lapse of time from the pulse onset. In the figure, simulation results are shown only for the curve (c) to avoid complication. The simulated temperature response (c′) is a little smaller than the measured one in this case. The



(a) Ethyl alcohol ($\bar{r}$=2.6x10^6 K/s, $\bar{q}_l$=8.78 MW/m^2)

(b) Ethyl alcohol ($\bar{r}$=10.7x10^6 K/s, $\bar{q}_l$=18.1 MW/m^2)

(c) Water ($\bar{r}$=5.26x10^6 K/s, $\bar{q}_l$=50.7 MW/m^2)

(d) Water ($\bar{r}$=37.7x10^6 K/s, $\bar{q}_l$=134 MW/m^2)

FIG. 5. Four photographic records of the aspect of boiling just after the incipience.

discrepancy between them is thought to be due to the error in evaluating the heat generation rate during the initial stage, where very large change occurs as shown in Fig. 3. The curve (c″) for heat flux shows that the value is almost constant except for the initial stage.

Figure 5 shows photographs indicating the changes in the aspect of boiling just after the incipience for the respective four conditions. It should be noticed here that a series of four frames under a condition are not successive during the same heat pulse but were chosen from ones of different heat pulses, where all other conditions except time are the same.

The aspect of boiling may be summarized as follows. The general tendency of the aspect is essentially the same for every liquid tested, although the dependency on the rate of temperature rise differs from liquid to liquid. When the rate is very low for each of the liquids in this experimental condition a single bubble generated on the heater grows and overspreads. At a low rate of temperature rise a few bubbles generate and grow as shown in Fig. 5(c). At a moderate rate of rise, as shown in Fig. 5(a), a number of bubbles generate and grow. When the rate of rise is high a large number of tiny bubbles generate concurrently on the heater in a short period as shown in (b) and (d) of Fig. 5.

Observing the frames in Fig. 5(c), it can be estimated that two bubbles on left-hand side may be generated from the nucleus already existing on the wall, because the two bubbles are generated repeatedly at the same positions in every frame. On the other hand, bubbles are not always generated at the same positions as the bubbles generated at the former stages in (a), (b) and (d) of Fig. 5.

Figure 6 demonstrates two frames taken from a photo under a condition of a high rate of temperature rise as well as a lapse in time, where the pulse shot is only different. Almost all bubbles, in one frame, are generated at different positions from those in the other. Thus, it is estimated that the nucleation is not due to those pre-existing sites on the wall but due to fluctuation nucleation.

Additionally, as observed near the voltage taps in Fig. 5(b), frame 4, there exist areas of no bubble generation on the heater. This may be caused by the fact that the area was not heated to the bubble generation temperature because of a small film thickness excess formed during manufacture.

When the rate of temperature rise is high, a large number of tiny bubbles whose diameters are nearly equal, are generated on the heater concurrently as shown in Fig. 5(b4) and (d4) for ethyl alcohol and water, respectively. The authors have published a brief report [14] on this type of bubble generation in ethyl alcohol and have named it 'caviarwise bubble generation', because it looks like 'caviar spread on bread'. In this experiment the same aspect was found to exist in water and toluene.

Observing Fig. 5(a) and (b4), which are for ethyl alcohol, one can recognize the area of no bubble generation near the side edges of the film heater. The area is narrower in water as shown in Fig. 5(d) and Fig. 6. To make clear the cause, a series of two-dimensional heat conduction simulations were conducted for the perpendicular plane to the heater axis. Figure 7 shows a typical simulation result and the temperature distribution near a side edge of the heater indicated with isotherms. The figure shows that the affected area is restricted to a small distance from the side edge when a higher rate of temperature rise is applied, as in water, but it reaches broader area with the lower rate. Temperature profiles on the heater wall and in the direction of heater width were obtained from a series of simulations such as those mentioned above. As a result, the area affected by the side edge was proven to almost correspond to the area of no bubble generation. Furthermore, the measured average temperature of the film heater was corrected with those simulation results to obtain the temperature at boiling incipience, which

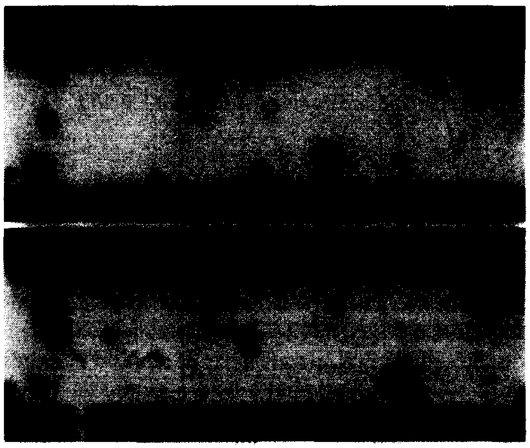


FIG. 6. Two frames taken under the same experimental conditions except only for the pulse shot (water, $\bar{r} = 48.0 \times 10^6$ K s^{-1}, $\bar{q}_1 = 152$ MW m^{-2}, $t = 5.25$ μs).

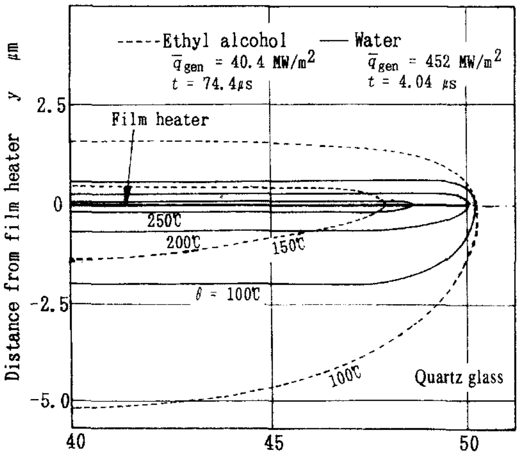


FIG. 7. Typical simulation results of temperature distribution near a side edge of the heater.

is the uniform temperature around the central part of the heater wall.

3.2. *Temperature at boiling incipience*

Figure 8 shows the temperature–time curves for five typical cases of the rate of temperature rise in ethyl alcohol. The temperature scale is changed at 195°C. The increasing tendency of the bubble number on the heater is also shown. The dotted line of $n = 1$ shows the condition at which the first bubble was observed and boiling occurred. The figure shows that a higher rate of temperature rise makes the temperature at boiling incipience higher.

In Fig. 9, the temperature at boiling incipience was plotted against the rate of temperature rise for ethyl alcohol, toluene and water, respectively. In every case the temperature at boiling incipience increases with the increase of the rate of the temperature rise and reaches a constant saturated value. The experimental results by Skripov *et al.* [6] using a fine wire in ethyl alcohol and water are plotted for comparison. Derewnicki [8] also used a fine wire heated with the rate of temperature rise up to about 10×10^6 K s^{-1} and obtained a value 20°C lower than the homogeneous nucleation temperature. It must be noted here that the aspect of boiling in the range where the temperature at boiling incipience is saturated becomes that of 'caviarwise bubble generation.'

The dotted line indicates the theoretical homogeneous nucleation temperature for each liquid, which is calculated from equation (1) in the next section, by assuming that one bubble nucleates on the test heater under atmospheric conditions. Although it increases a little with the rate of the temperature rise, it is almost constant. The experimental saturation value for ethyl alcohol agrees very well with the homogeneous nucleation temperature. Relatively good agreement is attained for toluene, although the experimental value is a little lower. On the other hand the saturated value for water is about 19°C lower than the theoretical one, which Skripov [4] has given 312.5°C as the theoretical value on the basis of the observed bubble number. The reason why such disagreement exists for water in spite of a good agreement for organic liquids is not clear at this stage: however, this may be due to the difference in wettability with the solid surface. A detailed discussion on this issue will be given in the next section.

3.3. *Considerations on the experimental results and fluctuation nucleation phenomenon*

The rate of formation of nuclei formed by fluctuation nucleation in a superheated liquid is given using the classical nucleation theory, including the one by Volmer [3] by way of an example. Equation (1) gives the rate per unit volume of a bulk liquid whose temperature is T; this case is called homogeneous nucleation. On the other hand, the nucleation on the bounding surface between two phases is called heterogeneous nucleation and the rate per unit surface is given by equation (2).

$$J_{\mathrm{homo}} = N_0\left\{\frac{6\sigma}{\pi m(3-b)}\right\}^{0.5} \exp\left\{-\frac{\lambda_v}{kT}\right\} \times \exp\left\{-\frac{16\pi\sigma^3}{3kT(p_s-p)^2(1-\rho_l/\rho_v)^2}\right\} \quad (1)$$

$$J_{\mathrm{het}} = N_0^{2/3}\psi\left\{\frac{6\sigma}{\pi m(3-\mathrm{b})\,\omega}\right\}^{0.5} \exp\left\{-\frac{\lambda_v}{kT}\right\} \times \exp\left\{-\frac{16\pi\sigma^3\omega}{3kT(p_s-p)^2(1-\rho_l/\rho_v)^2}\right\}, \quad (2)$$

where

$$b = 1-p/p_s, \quad \psi = \tfrac{1}{2}(1+\cos\phi),$$

$$\omega = \tfrac{1}{4}(1+\cos\phi)^2\,(2-\cos\phi).$$

In the present experimental system, homogeneous nucleation occurs within a very thin superheated liquid layer which has a very steep temperature gradi-

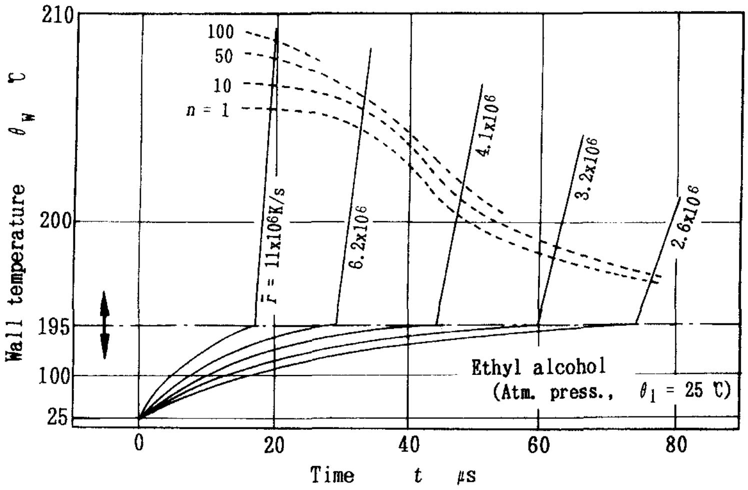


FIG. 8. Measured temperature–time curves in ethyl alcohol and increasing tendency of the bubble number.

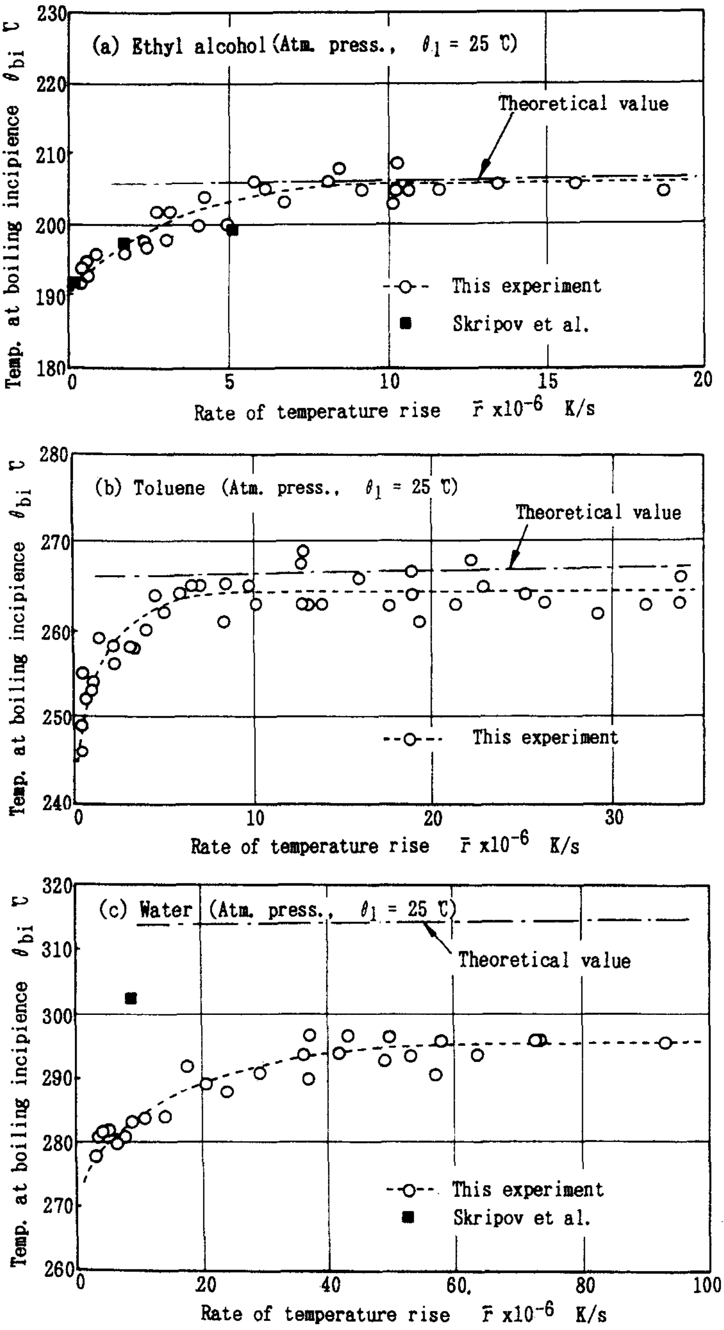


FIG. 9. Measured temperature at boiling incipience vs the rate of temperature rise for (a) ethyl alcohol, (b) toluene and (c) water.

ent, as shown in Fig. 7, and heterogeneous nucleation occurs on the heating wall. Since the phenomenon is observed from the upper side of the heater in this study, the number of nucleations, i.e., the number of observed bubbles in experiments, is given by equation (3) by assuming a linear temperature profile in the superheated layer.

$$N = \int_0^t \left\{ \int_0^{\infty} J_{\text{homo}}(\theta)\,\mathrm{d}x + J_{\text{het}}(\theta_w) \right\} \mathrm{d}t. \tag{3}$$

Figure 10 shows the calculation results of number density N and bubble number n on this heater wall vs the wall temperature for water. Equations (1) and (3) are used in this calculation and the contact angle on the wall is assumed to be zero. The conditions for the rate of temperature rise are selected from the analytical solution of unsteady heat conduction of pulsed heating and the numbers along the lines show the lapse of time after the wall temperature reaches 300°C. The figure shows that the bubble can be initially

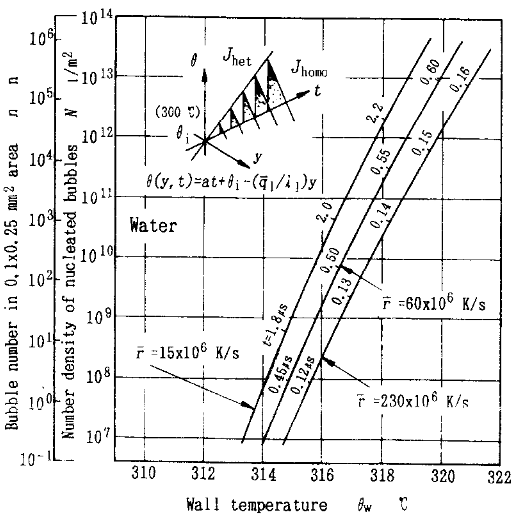


FIG. 10. Calculated homogeneous nucleation number vs wall temperature, rate of temperature rise and lapse of time (water).

observed when the wall temperature reaches about 314°C and that the number density reaches about ten times with a 1°C rise of the wall temperature. The reason why the lower rate of the temperature rise makes the number density so large may be that both the time elapsed under the nucleation temperature and the thickness of liquid layer in which nucleation occurs become large.

Figure 11 shows the calculation results based on equations (1)–(3) in which the contact angle is varied as the parameter. The dotted line is for pure homogeneous nucleation. The figure shows that the temperature at boiling incipience becomes considerably lower if the contact angle exceeds 30°. When it is 90° boiling incipience at a temperature lower than 300°C is predicted from the figure. This is considered to be one of the reasons why the temperature at boiling incipience is lower than the homogeneous nucleation temperature obtained for water, which was described in Section 3.2. This consideration agrees with the presumption by Derewnicki [8], in which boiling incipience at 20°C below the predicted homogeneous nucleation temperature may be attributed to the onset of spontaneous nucleation on a poorly wettable surface.

In Fig. 12, the bubble number observed on the heater immersed in water was plotted as a function of time for two rates of the temperature rise. In the case of a low rate, the bubble number increases to a constant value of about 20 after the boiling incipience at about 4.4 μs, and then bubbles grow steadily to aggregate. In the case of a higher rate, the bubble number increases very rapidly and reaches about 300, which is the maximum countable number. This is a case of the caviarwise bubble generation described previously. The solid line in the figure shows the theoretical value due to homogeneous nucleation under the previous experimental condition. The tendency of the both lines is roughly the same, although the experimental result is smaller than the theory. Assuming that nucleation is due to fluctuation nucleation only, the fact that a smaller number is obtained in experiments is estimated as follows: (1) the liquid volume served for nucleation decreases with time after boiling incipience and (2) latent heat consumption by boiling cools the surrounding liquid.

Figure 13 shows the bubble number vs the wall temperature for three rates of temperature rise in ethyl alcohol. The number increases rapidly after boiling incipience in every case. The solid lines are of the theoretical value of homogeneous nucleation number for two cases of the rate examined in the experiments. For $\dot{r} = 2.6\times10^6$ K s^{-1}, the number increases at a temperature lower than that of the theoretical one. On the other hand, the experimental and theoretical values are in good agreement for $\dot{r} = 11\times10^6$ K s^{-1} at which the caviarwise bubble generation occurs.

The maximum number densities of bubbles counted at respective conditions vs the rate of the temperature

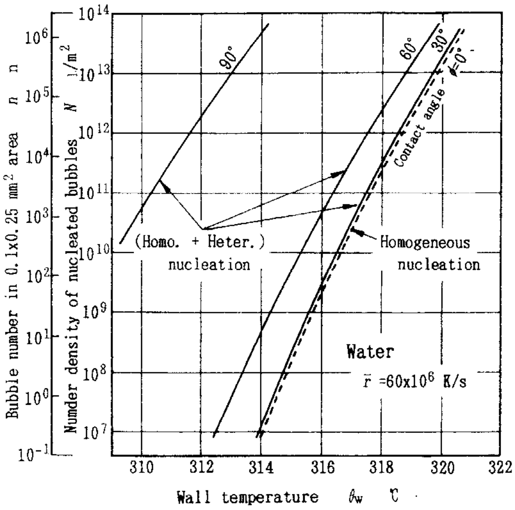


FIG. 11. Calculated homogeneous and heterogeneous nucleation number for various contact angles (water).

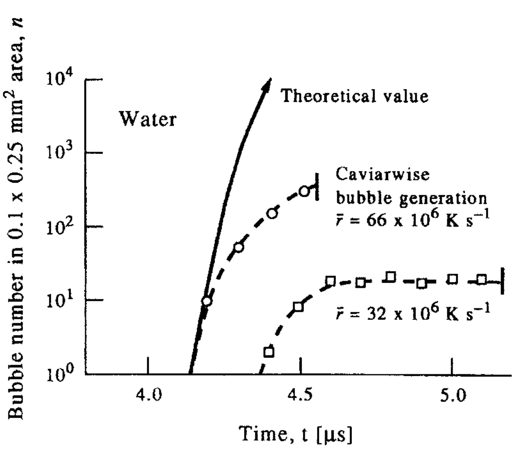


FIG. 12. Comparison between experimental and theoretical results for increasing bubble number for water.

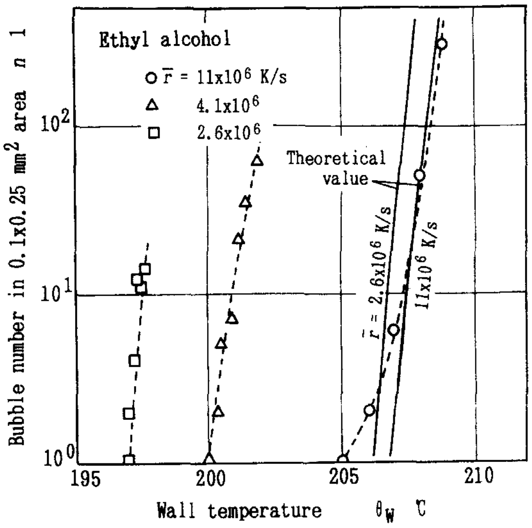


FIG. 13. Comparison between experimental and theoretical increasing bubble number for ethyl alcohol.

rise are plotted in Fig. 14 for the three liquids examined. The number density increases rapidly with the increase in the rate for each liquid and reaches 10^{10} m^{-2} or more at the maximum. The density is extremely large when compared with the maximum measured bubble site density of 10^6 m^{-2} in steady-state nucleate boiling[15], although they cannot be compared directly because of the difference in the physical meaning.

On the basis of the above experimental results and discussions, the authors postulate that the boiling phenomenon at a higher rate of the temperature rise in this study, especially caviarwise bubble generation, is mainly due to fluctuation nucleation. The reasons for this are summarized as follows: (1) the temperature at boiling incipience increases with an increase in the rate of the temperature rise and reaches a saturated value, (2) the saturated temperature agrees with the homogeneous nucleation temperature for ethyl alcohol and toluene, (3) although the saturated temperature in water is about 19°C lower than the homogeneous nucleation temperature, this disagreement may be due to heterogeneous nucleation, (4) the bubble number generated from different sites for every pulse shot increases at a higher rate of the temperature rise, (5) the increase in the bubble number during caviarwise bubble generation shows a similar tendency with the theoretical value of homogeneous nucleation. Furthermore, the authors do not insist that caviarwise bubble generation only is due to fluctuation nucleation. The rate of nucleation due to it will increase with the increase in the rate of the temperature rise and it will become dominant at a higher rate of the temperature rise as shown in caviarwise bubble generation.

4. CONCLUSIONS

For studying boiling nucleation phenomenon by a pulsed heating method a very small film heater on a quartz glass substrate was heated pulsewise in liquid and an extremely high rate of temperature rise of 93×10^6 K^{-1} s at the maximum was attained. Ethyl alcohol, toluene and water under atmospheric pressure were tested and the aspect of boiling as well as the number density of nucleation were observed. The main results obtained are as follows:

1. Temperature at boiling incipience increases with the increase of the rate of temperature rise and reaches a saturated value at a certain high rate. The saturated value agrees with the homogeneous nucleation temperature for ethyl alcohol and toluene and about 19°C lower for water.
2. When the rate of temperature rise is high caviarwise bubble generation, in which a large number of tiny bubbles with almost uniform diameter generated concurrently on the heater, is observed for all liquids.
3. On the basis of the fluctuation nucleation theory a series of simulations were made on the interrelation between the rate of temperature rise, wall temperature, lapse of time and the contact angle between

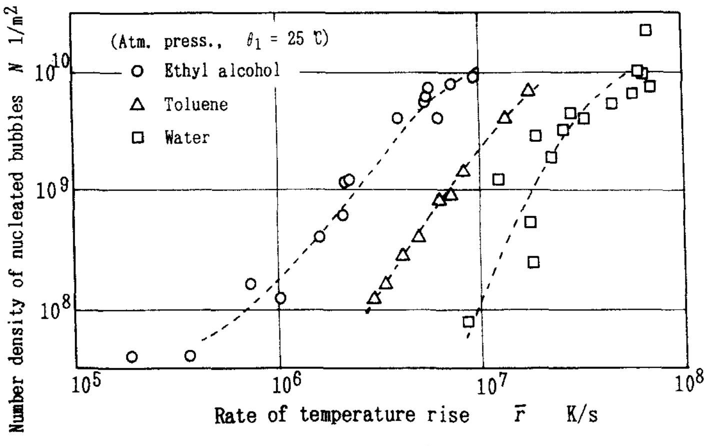


FIG. 14. Measured maximum number density of bubbles versus the rate of temperature rise.

liquid and wall surface. The results were compared with the experiment.

4. The number density of nucleated bubbles was plotted against the rate of temperature rise. The maximum density was 2×10^{10} m^{-2} in water.

5. The boiling nucleation at a higher rate of temperature rise, especially caviarwise bubble generation, is postulated to be due to fluctuation nucleation.

Acknowledgements—The authors would like to express their appreciation to Mr Junichi Sasaki, Mr Tarou Fukazawa, Mr Takahito Katayama and Mr Takehiro Nishizawa for their help with the experiments and computations. The authors would also like to thank the Ministry of Education, Science and Culture, Japan for the grant-in-aid for scientific research which partly supported this study.

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