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COMPARISON OF GAS AND VAPOUR BUBBLE NUCLEATION ON A BRASS SURFACE IN WATER

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Abstract—The size distribution of naturally occurring nucleation sites on a heater surface was determined by two experimental techniques—gas bubble nucleation as gas diffused out of a supersaturated solution and vapour bubble nucleation in boiling. Many more sites were active in gas diffusion than in boiling and the probable reasons for this are examined. The effective radii of individual sites were calculated from both sets of data. Finally, the sites were located and photographed by scanning electron microscope.

NOMENCLATURE

h_{fg}, latent heat of vaporisation;
N, number of active sites;
N_0, number of active sites for $\tau_i = 0$;
P, pressure;
P_A, pressure in vessel A;
P_B, pressure in vessel B (1, 2 referring to the initial and final pressures in vessel B during the prepressurisation experiments);
ΔP, $(P_A - P_{B2})$;
ΔP_p, $(P_{B1} - P_A)$;
r_e, effective radius of nucleation site;
T, temperature;
ΔT, $(T_W - T_{sat})$;
T_W, wall temperature;
T_{sat}, saturation temperature of the liquid;
v_{fg}, specific volume change during vaporisation.

Greek symbols
σ, surface tension, liquid/air;
τ_i, waiting period; the immersion time prior to pressure reduction at the test surface.

1. INTRODUCTION

THE EFFECT of surface conditions on nucleate boiling heat transfer can only be determined by experiment but it is often difficult or impossible to perform boiling experiments on surface samples of industrial interest, e.g. boiler or heat-exchanger tubes after in-service corrosion and fouling, engine cylinder liners; in other cases the range of variables to be examined may be impracticably wide. Thus there is still a strong incentive (besides the intellectual challenge) to develop a theoretical model which, perhaps in combination with a limited number of simple inexpensive experiments, can predict with reasonable accuracy the nucleate boiling performance of a given surface with any fluid for a specified pressure–temperature history.

All theoretical models of nucleate boiling need input data on the size distribution of cavities which are active as nucleation sites for a given fluid–surface combination. At present this data can only be obtained by experiment. Visual observation is successful only at low heat flow or high subcooling since vapour production obscures the surface; salt deposition or electroplating during boiling may change the surface characteristics; miniature voidage probes can be used up to high heat fluxes [1] but involve a tedious scanning process and are suitable only for water. Brown [2], working in the Heat Transfer Laboratory, Massachusetts Institute of Technology, suggested that population data for vapour bubble nucleation sites could be obtained by studying bubble nucleation from supersaturated gas solutions. The attraction of this technique is its simplicity: the much slower growth rate of gas bubbles makes it easy to count nucleation sites visually or from photographs even at supersaturations corresponding to high superheats. However, it is first necessary to demonstrate the validity of the method by direct comparison with boiling at low superheats. The technique was developed by Lorenz, Mikic and Rohsenow [3], who obtained close agreement between size estimates from pool boiling, gas nucleation and microscopic measurements for individual cavities on a copper surface with water. The same cavities had smaller effective radii during boiling of organic fluids, indicating an important effect of contact angle. (It has also been suggested [4] that inertia effects may be significant for organics during liquid entry into the cavity.) The gas bubble technique could not be applied to organic liquids because of the rapid bubble growth rate at atmospheric pressure.

In the M.I.T. technique, the surface under investigation is mounted in the bottom of an open channel. Liquid saturated with gas in a vessel at elevated pressure flows through a pressure-reducing valve and along the channel. Gas escapes from the solution at the free surface and by adjusting the flow

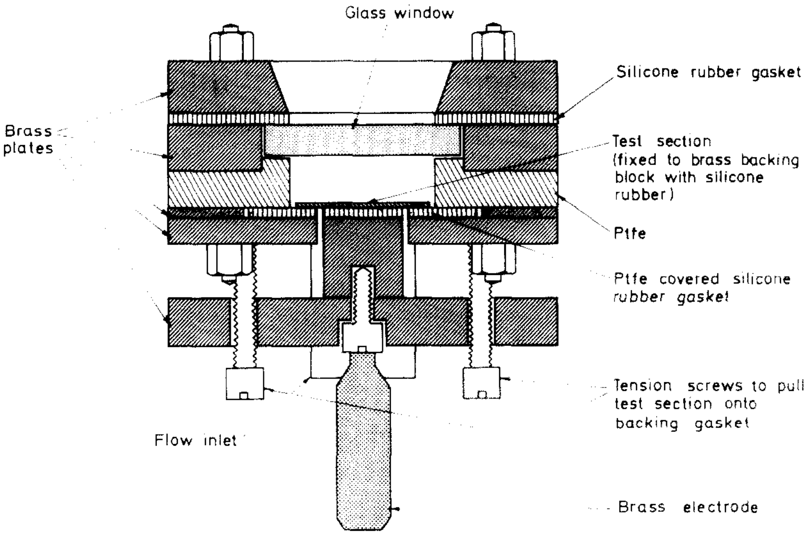

FIG. 1. Test section container.

rate the concentration at the test surface can be controlled in a continuous manner. The need to monitor local gas concentration and to operate at atmospheric pressure has restricted the method to solutions of air (O_2 measured) in water. Further, the good agreement with boiling was only achieved by treating the surface with soap and this throws some doubt on the method for investigating contact angle effects.

In the present study we have used a method of producing bubble nucleation from gas solutions which avoids the limitations of the M.I.T. method. The pressure reduction is performed in a closed system which can operate at pressures up to 8 bar. The gas concentration is uniform so local monitoring is unnecessary but the degree of supersaturation can only be increased in steps, instead of continuously. The ability to vary the pressure at the test surface permits the simulation of history effects in boiling, changes in system pressure or subcooling prior to nucleation. So far, experiments have been confined to water with nitrogen but we expect the method to be applicable to other fluids and gases. The excessively vigorous evolution of gas from supersaturated organic liquids [4] should be avoidable by operating at elevated pressures.

The test surface is mounted in a closed channel which can be incorporated either in the gas nucleation apparatus or in a flow boiling loop. In this paper we describe a detailed investigation of gas bubble nucleation on a brass surface (80% Cu, 16% Zn, 1% Ni, 1% Al), a comparison with nucleation sites in boiling on the same surface and finally the identification and examination of individual sites by scanning electron microscope.

2. EXPERIMENTAL METHOD

The test surface was cut from a 0.8 mm thick rolled strip of brass alloy. The strip was used as received, with no surface treatment other than careful cleaning. The surface was cleaned initially with acetone, then a hot detergent solution and finally rinsed with copious quantities of distilled water.

The test surface forms one wall of a rectangular channel, (Fig. 1). Electrodes are attached 205 mm apart for DC electrical heating. The surface temperature is measured by calibrated Cu–Con thermocouples attached to its rear by a thin layer of epoxy adhesive; electrical guard heating is provided. Flow connections, either to the flow boiling loop or to the gas nucleation rig, are 305 mm apart at right angles to the axis of the channel. Apart from the test surface, the only materials in contact with the liquid are glass, PTFE and stainless steel.

The flow boiling loop is a standard closed circuit containing pump, flow meter, electrical preheater, throttle valve, test section with vertical upward flow and a water-cooled heat exchanger. A separate vessel with an electric immersion heater is used for degassing and pressurising the system. The water is deionised, double-distilled to remove surface-active contaminants and placed in the rig within a few hours of distillation. It was found that prolonged storage in glass vessels led to surface deposits during boiling. The boiling experiments were performed at a pressure of 1.17 bar, water 3 K subcooled at test section inlet and a velocity of 0.15 m/s. Poor matching between the power supply and the low-resistance test surface restricted the maximum heat flux to 1.1×10^5 W/m^2 at the maximum superheat of 12.8°C. Bubble nucleation sites were conveniently located from cine films taken at 24 frames/s.

The gas nucleation rig is shown schematically in Fig. 2. Nitrogen is supplied to vessel A through a precision pressure regulator. The pressure P_A is measured by a Bourdon test gauge, or by mercury or water manometers at low pressures. Vessel A is half filled with water, which is saturated with nitrogen by recirculation through a spray nozzle for 45 minutes, after which it is allowed to settle for a further 30 min. The

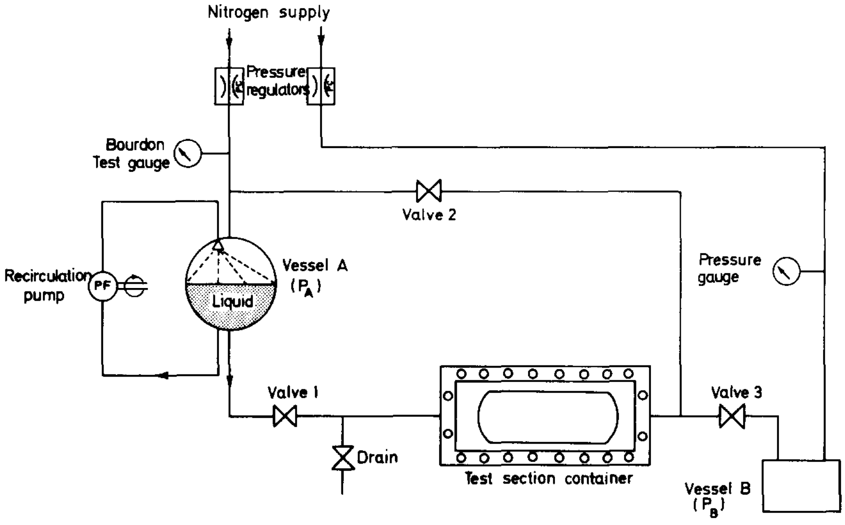

FIG. 2. Gas diffusion rig.

water temperature is kept at 25°C by a water-cooled heat exchanger on the pump circuit and an electrical immersion heater in the vessel. The test section is mounted horizontally 70 mm below the liquid level in A. It is connected to the liquid space of A by valve 1 and to the gas space by valve 2. In order to perform a test, valve 2 is opened and then valve 1 is slowly opened so that saturated liquid fills the channel and the pipe rising towards valve 2. Valve 1 is closed and the liquid left in contact with the test surface for an immersion time τ_i ranging from 5 s to several hours. Valve 2 is then closed and valve 3 opened, connecting the test section to vessel B, which is maintained at a lower pressure P_B by another precision regulator. Since the temperature and the dissolved gas concentration are constant, the liquid in the test section is now supersaturated by an amount corresponding to $\Delta P \times P_A - P_B$ and nucleation sites with effective radii $r_e > 2\sigma/\Delta P$ (1) are activated. Bubbles grow to 0.1 mm radius in typically 1–2 min and are then photographed with a 35 mm camera focussed on a rectangle 18 × 12 mm. The corners of the rectangle are marked with fine scratches so that the coordinates of individual nucleation sites can be obtained to ±0.05 mm. The test section is finally drained, heated to 35°C by passage of a low electric current and thoroughly dried in a stream of nitrogen ready for the next test at a different ΔP.

The pressure reduction ΔP is readily converted into an equivalent superheat in boiling. Liquid in a boiling system may initially be subjected to subcooling, so that the system pressure exceeds the vapour pressure. This condition is simulated in the gas nucleation rig by initially setting the pressure in vessel B to a value P_{B1} higher than P_A before dropping it to the lower value P_{B2} to cause nucleation. (There is a static column of liquid 10 cm long between the point of application of P_B and the test section so the gas concentration in the test zone is unaltered.) This prepressurization causes a considerable reduction in the number of nucleation sites for a given $\Delta P = P_A - P_{B2}$ and is an important step in identifying nucleation sites which are stable in boiling.

The actual analysis of the gas diffusion data is done from slides of the 35 mm photographs. The fine scratches on the test surface provide the reference points. A slide is projected onto a screen of squared paper and the positions of the gas bubbles marked. These can then be compared with any other set by projecting a second slide onto the same screen.

3. GAS BUBBLE NUCLEATION

3.1. *General*

Gas nucleation experiments produce information at two levels: (i) details of the characteristics of individual cavities, (ii) overall population data, the size distribution curve. For the purpose of testing the relationship between nucleation from gas solutions and in boiling it is instructive to examine individual sites. If a relationship can be proved, the gas nucleation technique will be a convenient way of obtaining population data for different fluid–surface combinations and examining variability between samples of the same surface type.

3.2. *Tests without prepressurisation; reproducibility*

Preliminary tests showed that reproducible results could only be obtained by thoroughly drying the surface between each test, as described in Section 2. The immersion time τ_i affects nucleation, the bubble population decreasing to a constant value for immersion times exceeding 10 min, Fig. 3. All the tests reported below were performed with immersion times of 20 min; in subsequent tests with prepressurisation the excess pressure was applied for 20 min immediately after immersion.

Tests were performed for four different pressure drops, each test being repeated four times. The results are summarised in Table 1 and Fig. 4 ($\Delta P_p = 0$). For a given ΔP, the overall bubble population did not vary more than ±6%. Individual nucleation sites showed a somewhat greater variability with 80% of the sites appearing in all four tests at $\Delta P = 0.27$ bar and 88%

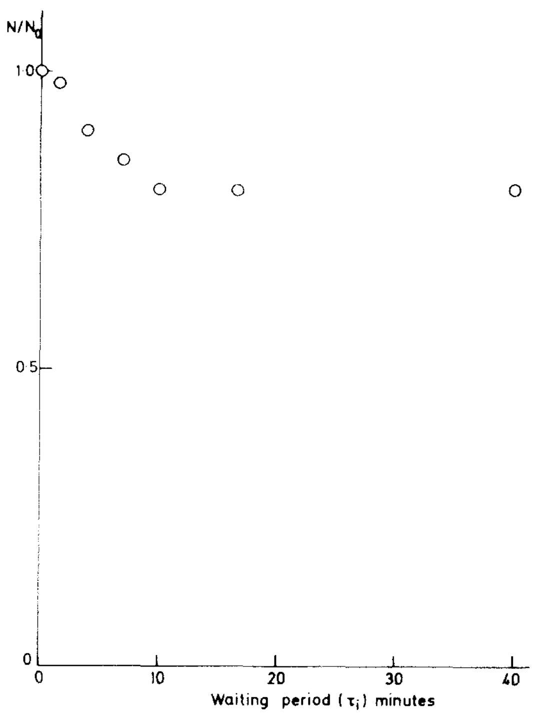

FIG. 3. Effect of waiting period on bubble population.

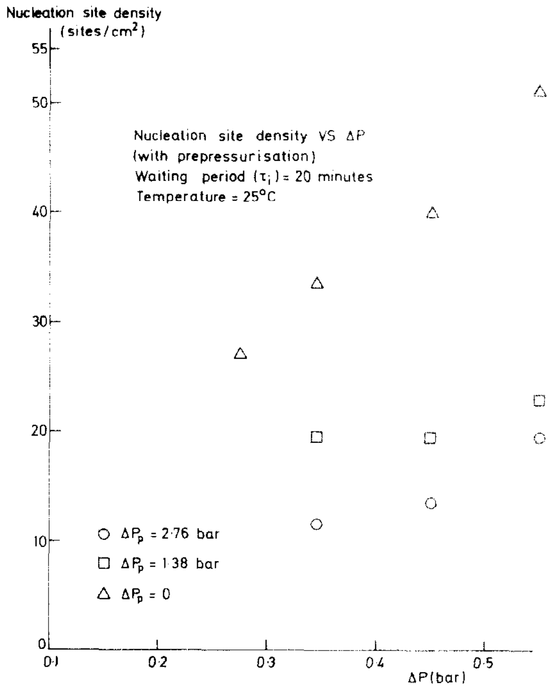

FIG. 4. Effect of prepressurisation on bubble population.

Table 1. Gas bubble nucleation, no prepressurisation

ΔP bar	Total number of sites, area 2.16 cm^2	Number of sites appearing 4, 3, 2, 1 times	Total number activated
0.55	107, 100, 102, 101	90, 10, 8, 4	112
0.45	83, 78, 80, 75	68, 10, 6, 2	86
0.34	63, 68, 63, 64	56, 6, 7, 2	71
0.27	52, 52, 56, 52	46, 6, 4, 2	58

appearing in all four tests at $\Delta P = 0.55$ bar. Those sites which appeared intermittently were invariably the smaller members of the group, with an activation ΔP close to the experimental ΔP. One source of variation is the experimental uncertainty of ± 0.02 bar in repeating the same nominal pressure drop: from Fig. 4 this could account for ± 8% variation in population at the lowest ΔP. Also cine films of the liquid flooding the test surface showed variations of up to 45° in the orientation of the interface from run to run. As naturally occurring nucleation sites are unlikely to be symmetrical this may affect the initial trapping of gas and hence r_e.

3.3. *Prepressurisation*

The effect of prepressurisation is to reduce the bubble population, Fig. 4. A detailed analysis of the 31 sites eliminated by prepressurisation at $\Delta P = 0.34$ bar was made to see if they were reactivated by larger values of ΔP. The 31 sites fell into two groups:

Group (*i*) 22 sites which did not survive $\Delta P_p = 1.38$ bar.

Group (ii) 9 sites which did not survive $\Delta P_p = 2.76$ bar but did survive $\Delta P_p = 1.38$ bar.

The number of sites from each group which appeared at higher pressure drops is given in Table 2.

Table 2. Behaviour of 31 sites affected by prepressurisation at $\Delta P = 0.34$ bar.

ΔP_p(bar) / ΔP(bar)	0		1.38		2.76	
0.34	22	9	0	9	0	0
0.45	22	9	4	9	0	2
0.55	22	9	5	9	0	6
Group	(i)	(ii)	(i)	(ii)	(i)	(ii)

Only a quarter of the sites in group (i) were activated by the maximum ΔP of 0.55 bar after prepressurisation by 1.38 bar and none was active after prepressurisation by 2.76 bar. Of group (ii), two thirds could be activated at the maximum ΔP. Thus for group (ii) the effect of prepressurisation was to drive nuclei to equilibrium positions with smaller values of r_e. Without information on internal geometry it is not possible to say whether this also applies to group (i), or whether prepressurisation caused complete wetting and deactivation. Also it must be remembered that for many sites prepressur-

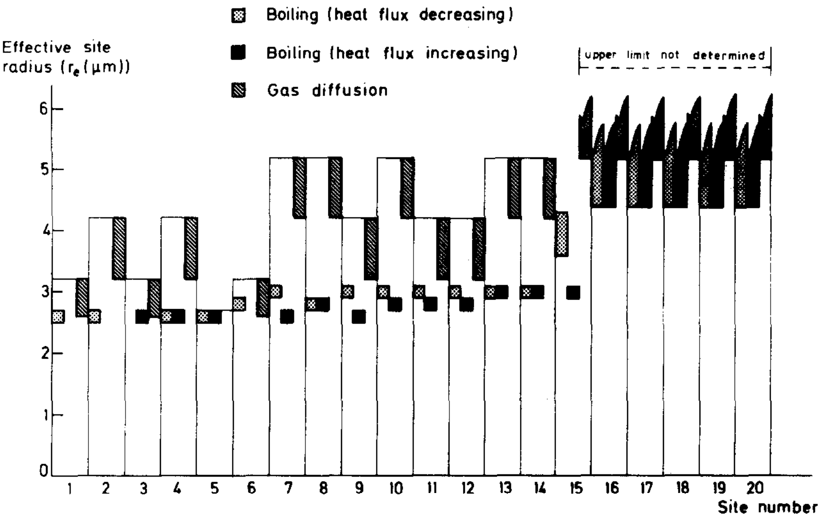

FIG. 5. Comparison of site sizes for the different nucleation modes.

Table 3. Site classification

A. (1), (3), (6)	reasonable agreement
B. (2), (4), (9), (11), (12)	gas approximately 40% too large
C. (7), (8), (10), (13), (14)	gas approximately 65% too large
D. (16), (17), (18), (19), (20)	gas approximately 20% too large, but note that upper limit is not determined.
E. (15)	boiling hysteresis pronounced, good agreement with gas for deactivation

isation did not change r_e, this group including the sites which were later found to be active in boiling.

4. COMPARISON WITH BOILING

On completion of the gas nucleation tests, the entire test section was transferred to the flow boiling rig for boiling experiments with degassed water.

Boiling took place for one hour at the maximum heat flux attainable, 11 × 10^4 W/m^2. The heat flux was then reduced in steps of about 1.5 × 10^4 W/m^2–2.2 × 10^4 W/m^2, the lowest flux at which boiling occurred. After boiling for 30 min at each heat flux, 15 s of cine film were shot and the heat flux then decreased to the next value. Identical 'steps' were then taken back up to the maximum heat flux and film was taken on each occasion.

Wall temperatures were measured by four thermocouples attached to the back of the test strip behind the reference 18 × 12 mm rectangle. The readings were corrected for temperature gradient through the wall; temperature gradient effects in the liquid were negligible because of the small cavity sizes and low heat fluxes.

In all, 20 active boiling sites appeared in the region of measurement. Their location could be determined to ±0.5 mm from a frame of the cine film showing the early stages of bubble growth. This was less accurate than the gas bubble site determination since bubbles were displaced downstream slightly as they grew to their departure radius of approximately 1 mm. For each site the effective radii for activation and deactivation were determined from the wall superheat ΔT using

$$r_e = 2\sigma T_{\text{sat}} v_{fg} / h_{fg} \Delta T \tag{2}$$

and compared with the value from gas diffusion, Fig. 5. The calculated value of r_e in both gas diffusion and boiling is limited by the ΔP and ΔT increments chosen during the experimental program.

Boiling hysteresis produced some scatter in the estimates of site radius. However, hysteresis in boiling was less than 10% r_e for all sites except (15).

An examination of Fig. 5 shows that the sites fall into the following classes when estimations based on gas diffusion data are compared with those from boiling (Table 3).

Only one of the 20 sites was active in boiling but not in gas diffusion. Eighteen sites were active in gas diffusion at a prepressurisation of 1.38 bar, 17 at ΔP_p of 2.76 bar. For these sites, the effective radius was unaffected by the amount of prepressurisation. However, there were many sites active in gas diffusion and stable even under prepressurisation which were not active in boiling, Fig. 4. For zero prepressurisation the gas site density was about 5 times that in boiling, falling to twice the boiling value at $\Delta P_p = 2.76$ bar. This inactivity of apparently stable sites must be explained if the results from gas diffusion experiments are to be used to predict boiling curves. One possibility is thermal interference from adjacent active sites: the bubble departure radius in the boiling experiments was approximately 1 mm, indicating a circle of in-

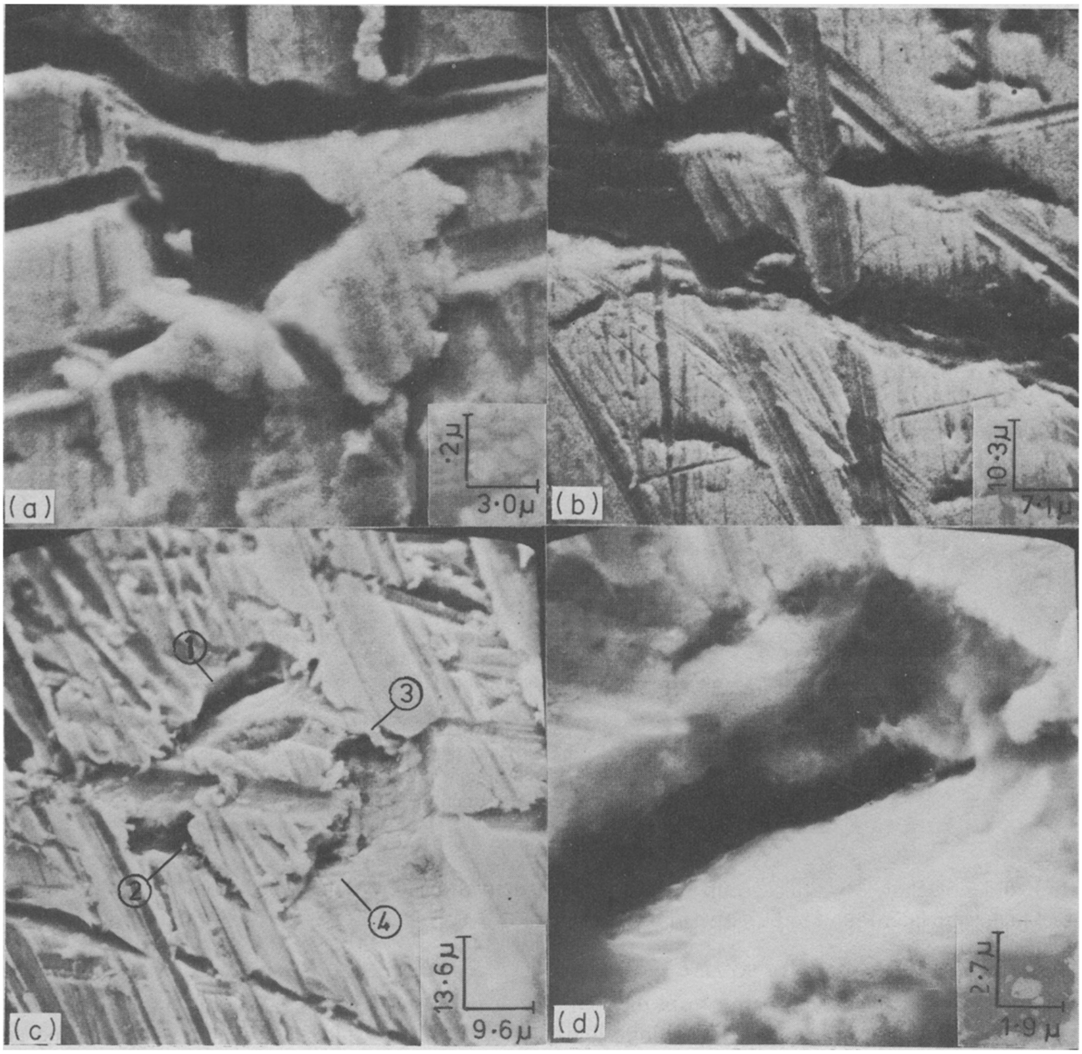

FIG. 6. (a) Sharp edged pit site No. 6, $2.6 \leqslant r_e \leqslant 3.2\mu$.
(b) Part of crevice containing site No. 4. $3.2 \leqslant r_e \leqslant 4.2\mu$. Precise position not known.
(c) Candidate sites in area containing site No. 3. $2.6 \leqslant r_e \leqslant 3.2\mu$.
(d) Probable active site (1) in (c), since (2), (3) and (4) smaller than r_e.

fluence of say 2 mm radius [5]. Of the 21 sites which were active in gas diffusion experiments at $\Delta P_p = 2.76$ bar but not in boiling, 19 were found to lie within the circle of influence of an active site of larger effective radius. This supports the thermal interference hypothesis but further experiments are required e.g. using changes in flow rate or subcooling to modify bubble departure size.

5. NUCLEATION SITE GEOMETRY

Since the typical length scale of a nucleation site is a few microns, magnifications of approximately 10^3–10^4 are required for detailed microscopic examination. The scanning electron microscope (SEM) has a limited field of view at these magnifications, making it difficult to find individual sites. The rather large bubble departure size in boiling, coupled with possible asymmetry relative to the nucleation site produces uncertainty in the site position. By using the much more precise positional data from the gas diffusion experiments it was possible to locate and photograph most of the active nucleation sites.

In half the cases there was more than one candidate pit or scratch in the estimated region of the nucleation site. Pits or scratches with a maximum dimension less than the effective diameter of the site were eliminated. In all, 16 of the active boiling sites were identified with reasonable certainty. In the other four cases it was impossible to identify one particular cavity as the actual site. In 11 cases the active cavities were sharp edged pits on the surface. The other five sites appeared to lie in long crevices, sometimes 2–3 times wider than the calculated value of site diameter. There was nothing to suggest that grain boundaries formed active nucleation sites. Two sites which survived prepressurisation but did not nucleate during boiling were also examined. They did not appear to differ physically from those sharp edged pits which were active during boiling.

Some of the sharp edged pits were not unlike a number of the sites identified by Nail, Vachon and Morehouse [11] on a stainless steel surface. However, some of their sites lay at grain boundaries, something which did not occur on the brass surface examined here.

Figure 6(a) shows a typical sharp edged pit site, while Fig. 6(b) is part of a crevice which contains an active site. Figure 6(c) shows an area which contains an active site and Fig. 6(d) is a close-up of what is thought to be the actual site in this area. Maximum resolution is obtained in the SEM when the specimen is at 45° to the incident beam. This produces a shortening of the vertical dimensions as viewed in the photographs.

The values of r_e as calculated from the gas diffusion and boiling data were closest when the nucleation site was a sharp edged pit. All the class A sites (Table 3) and four of the class D sites were pits, the fifth class D site could not be clearly identified. The five sites of the crevice type were all class B and C sites—the classes which showed the greatest discrepancies in r_e values. There was one pit site in each of these classes too, so any conclusions as to the relative behaviour of these two different types of sites should reflect this discrepancy. It has been impossible so far to investigate the internal shape of the cavities, although some of the scanning electron microscope photographs do provide partial views of the interior. This information may be obtainable by a surface replication technique currently being examined.

6. CONCLUSION

For water on rolled brass strip, our experiments have shown that the gas diffusion technique gives estimates of effective cavity radius ranging from good agreement with boiling to 65% larger. This is a greater variation than that obtained by Lorenz, Mikic and Rohsenow [3]. However, they first treated their surface with a surfactant, suggesting that the effects of variation in contact angle require further investigation. In our admittedly small sample the best agreement between gas diffusion and boiling was for sharp-edged cavities, which might be less sensitive to the precise value of contact angle than cavities with less well defined mouth geometry. The importance of contact angle to the initial trapped volume (and hence r_e) has already been demonstrated by Bankoff [6]. Contact angle is sensitive to temperature [7], the velocity of the interface [8, 9] and the relative humidity of the gas phase[10]. Deposition of solids during boiling may also change the contact angle and surface geometry in the vicinity of active sites.

Prepressurisation provides a good test for site stability but many of the stable sites in gass diffusion are not active in boiling. Our experiments suggest that allowance for thermal interference between sites may improve the correlation between site densities in gas diffusion and boiling but further investigation is required. If thermal interference is important, the interaction between site density and maximum bubble size emphasises the need for an improved model for bubble departure.

There was no evidence in these experiments of activation of unstable sites by adjacent stable sites, but more recent measurements on a stainless steel surface at higher heat fluxes suggest that both thermal interference and seeding of unstable sites are important [12].

The gas diffusion technique shows promise as a predictive method for nucleate boiling but needs further testing on a range of surfaces, which may have different proportions of stable and unstable sites and of the various cavity geometries identified in this study.

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COMPARAISON DE LA NUCLEATION DES BULLES DE GAZ ET DE VAPEUR SUR UNE SURFACE DE LAITON DANS L'EAU

Résumé—On a déterminé la distribution en taille des sites naturels de nucléation, sur une surface chaude, par deux méthodes expérimentales: la nucléation de bulles de gaz qui diffusent hors du métal et la nucléation des bulles de vapeur dans l'ébullition. Beaucoup plus de sites sont actifs en diffusion gazeuse qu'en ébullition et on examine les raisons probables. Les rayons effectifs des sites individuels sont calculés dans les deux cas. Les sites sont localisés et photographiés à l'aide d'un microscope électronique.

VERGLEICH VON GAS- UND DAMPFBLASENBILDUNG AN EINER MASSINGOBERFLÄCHE IN WASSER

Zusammenfassung—Die Größenverteilung von natürlichen Keimstellen an einer Heizwand wurde mit zwei experimentellen Methoden bestimmt: Gasblasenbildung bei der Diffusion von Gas aus einer übersättigten Lösung und Dampfblasenbildung beim Sieden. Bei der Diffusion von Gas waren mehr Keimstellen aktiv als beim Sieden. Die Gründe hierfür werden untersucht. Die effektiven Keimstellenradien wurden aus den Ergebnissen beider Versuchsreihen berechnet. Der Ort der Keimstellen wurde mit einem Elektronenmikroskop fotografisch bestimmt.

СРАВНЕНИЕ ПРОЦЕССОВ ЗАРОЖДЕНИЯ ГАЗОВОГО И ПАРОВОГО ПУЗЫРЬКОВ НА ЛАТУННОЙ ПОВЕРХНОСТИ В ВОДЕ

Аннотация — Распределение по размерам естественных центров зарождения пузырьков на поверхности нагрева определялось двумя экспериментальными способами: образование газовых пузырьков при диффузии газа из сверхнасыщенного раствора и образование пузырьков пара при кипении. В первом случае наблюдалось гораздо больше центров, чем во втором. Проведен анализ возможных причин этого явления. Для этих двух случаев рассчитывались эффективные радиусы отдельных центров. Наконец, местоположение центров определялось и фотографировалось с помощью сканирующего электронного микроскопа.