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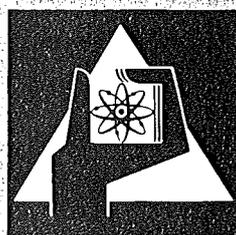
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Institut für Reaktorentwicklung

**Hypothesis about Nucleation Boiling Events
Occuring at Phase Boundaries**

G. Class, G.F. Schultheiß



**GESELLSCHAFT
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Hypothesis about Nucleation Boiling Events
Occuring at Phase Boundaries

G. Class

G. F. Schultheiss

Gesellschaft für Kernforschung mbH, Karlsruhe

Hypothese zur Keimbildung bei Siedevorgängen an Phasengrenzflächen

Zusammenfassung

Zur Erklärung der bekannten Erscheinung, daß die Bildung von Siedekeimen in heterogenen Systemen stark begünstigt ist, werden die Benetzungsverhältnisse im Mikrorißsystem im Bereich der Festkörperoberflächen mit herangezogen. Innerhalb dieser Mikrorisse, die entweder vorhanden sind oder während des Prozesses entstehen, verändert sich der Benetzungswinkel zwischen fester Wand und Flüssigkeit prozeßbedingt und kann 90° erreichen und überschreiten. Für dieses Verhalten werden örtliche Adsorptions- und Absorptionsprozesse verantwortlich gemacht.

Zusammen mit dem Effekt der spontanen Keimbildung im submikroskopischen Bereich entsteht so ein hypothetisches Keimbildungsmodell mit dynamischen Eigenschaften, besonders hinsichtlich der Existenz der einzelnen Keimstellen. Es erlaubt, das statistische Verhalten der makroskopischen kritischen Dampfkeime zu deuten (wie Siedeverzugs- und Wartezeitschwankungen).

Abstract

Wetting conditions in a system of microcracks in the range of solid surfaces are proposed as a basis for explaining the familiar phenomenon that the generation of boiling nuclei is greatly favored in heterogeneous systems. Within such microcracks, which either exist or are generated in the process, the wetting angle between the solid wall and the liquid is influenced by the process itself and can reach or even exceed 90° . Local adsorption and absorption processes are believed to be responsible for this behaviour.

This together with the effect of spontaneous nucleation in the sub-microscopic range gives rise to a hypothetical model of nucleation with dynamic characteristics, especially with respect to the existence of individual nucleation sites. In this way, the statistical behaviour of macroscopic critical vapor nuclei (such as variations of boiling superheat and delay times) can be explained.

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Introduction

A study of the theoretical and experimental work so far conducted with respect to nucleation events, e. g. /1, 2, 3/, to mention just a few summary reports, shows that the theory by Volmer, Weber, Becker and Döring has been applied above all to processes of condensation and cristallisation. Nucleation in connection with boiling events is treated only as a sideline.

In experimental studies, for which an optimum precision of all experimental parameters is very important, minute impurities at the surface of a solid (adsorption and absorption) and the generation of disturbing microcoatings etc. play such an important part that it is hardly possible to establish all the important parameters as accurately as they ought to be. Moreover, boiling processes occur chiefly in technical systems where they are influenced by other secondary effects; in this way, unequivocal statements on the basis of "purer" experiments with respect to various influences upon the mechanism of nucleation and liquid superheat are greatly aggravated if not rendered impossible at all.

Therefore, familiar and physically plausible model concepts of boiling nucleation are based on cavities in the surface of a solid primarily filled with gas, for instance in the heated wall of a system /4, 5, 6/. However, this model concept, which is used mainly for water and organic liquids, is very difficult to maintain for liquid alkali metals as those which will be used for heat transfer purposes in nuclear energy generation systems /7/.

Both under clean laboratory conditions and in technical systems, liquid metals show a marked tendency towards high liquid superheat. Besides the thermodynamical processes, numerous temperature dependent physico-chemical reactions can be observed because of the great chemical aggressiveness of this liquids at their high boiling temperatures. Also in water it has been found many decades ago, that it is possible experimentally to

achieve considerable superheat under special conditions /8/.

The possibility of suppressing the generation of an initial boiling nucleus in a larger range of conditions of a system has not been taken into account in the model setups known. There is only one model covering the inactivation of boiling nuclei during the boiling process /9/. Model concepts and hypotheses trying to explain the first generation of a boiling nucleus must also contain the possibility of a theoretically satisfactory explanation of the suppression of initial nucleation, and even nucleation proper, during this boiling process. Any question along these lines can be used as an additional criterion for checking models on the nucleation associated with boiling at phase boundaries.

In the following study a new hypothesis on the nucleation in boiling events at phase boundaries is presented. This hypothesis is based on generally known and accepted physico-chemical principles.

Hypothesis about Boiling Nucleation

Both, experimental experience and theoretical calculations /1, 2/ show, that the probability of critical nuclei of a new phase forming in a homogeneous phase is extraordinarily small. Hence, for real boiling events, it follows, that energetically more favourable processes must be possible in heterogeneous areas of a system (for instance at the heat transferring solid wall) than in the case of a homogeneous nucleation in order to explain the experimentally observed values of liquid superheat and the nucleation rate. The following hypothesis is developed for this purpose:

Nucleation in boiling processes originates from the very finest branchings of a system of microcracks covering the surfaces of solids.

Within these microcracks, which are present or are generated during the process, the wetting angle between the solid wall

and the liquid changes as a function of the process and can reach or exceed 90° , this may be influenced by geometrical effects.

In the range of dimensions of microchannels between $5 \cdot 10^{-10}$ and $12 \cdot 10^{-10}$ m there is spontaneous nucleation also below the normal boiling point at a sufficient frequency, which can be varified by the application of the theory by Vollmer et al. /1, 2/.

At wetting angles larger than 90° the bubble nuclei do not collapse again and continue to grow as a function of the wetting angle, the vapour pressure and the geometry of the microcracks. Over a certain period of time, this may cause the microcrack system to be increasingly drained of liquid even below the boiling temperature. If it had been filled with a liquid previously, it will then fill with vapour.

The nucleus may grow as a function of the random geometrical characteristics of the microcrack system and, in particular, of the changes in the wetting angle close to the openings in the crack system to the outer surface. When the boiling point is exceeded, nuclei generated in this way will have the same effect as the gas nuclei assumed in the wellknown model concepts. However, because of their different mechanism of generation, they react differently to changes of state of the system than do gas nuclei.

Explanation and Discussion of the Hypothesis

To provide a better understanding of the hypothesis covering nucleation in connection with boiling events at phase boundaries summarized above, the following ranges and events will be explained and discussed below:

The range of microscopic nucleation.

The transfer to the macroscopical critical vapour nucleus.

The statistical behaviour of the macroscopical critical vapour nuclei.

Within these crudely outlined areas a number of physico-chemical processes will occur either parallel or consecutively which depend on the state of the entire system and, in addition, are subject to statistical fluctuations.

The Range of Microscopic Nucleation

Metallic surfaces wetted by a liquid are subjected to continuous changes in heat transfer systems. During operation, the material will corrode or erode, and physico-chemical processes will result in deposition of surface coatings; rapid temperature cyclings may cause microcracks to be generated at the grain boundaries or crystals. Only in rare cases will these changes proceed slowly enough to allow the surface condition to be regarded as unchanged. Most of these continuous changes of the surface originate at defects generated in the working of the material. Fig. 1 gives an impression of a microstructure of a stainless steel surface worked by filing. Even finer methods of finishing produce surfaces covered with an extensive system of microcracks. Fig. 2 shows that such microcracks cannot even be removed by solution corrosion /10/. The existence of such microcrack systems on the surfaces of metals makes the true surface of metallic objects bigger than the directly visible surface. The extent by which the true surface becomes larger than the visible surface as a function of the type of working. This fact has been proved repeatedly by adsorption experiments /11/.

If physico-chemical processes are to occur also within such systems of fissures and microcracks of surfaces wetted by liquids, the wetting behaviour of the liquids must be sufficiently good, and the wetting angle must be below 90° . The liquid alkali metals initially regarded here, such as sodium, are known to present an excellent macroscopic wetting behaviour for temperatures above 400°C . A more accurate study conducted on liquid sodium /12/ indicated, that even microcracks can be filled with the liquid which, in this way, can penetrate into metal surfaces to a depth of several thousands of atomic

diameters. In this way, a physico-chemical change of the outer and inner surface within one layer becomes possible, whose thickness is equal to the depth of penetration of the liquid into the microcracks.

In the light of these considerations there may well be microcracks causing wetting angles in excess of 90° because of their surface structure, which are, therefore, not filled with the liquid. This links up directly with the theory by Holtz and Singer /17/; however, this theory supposes only such cavities to be the causes of nucleation.

Under the hypothesis advanced here it is possible that nucleation will start at microcracks, which are initially filled completely with liquid. Physico-chemical reactions may change the wetting behaviour of the (inner) surfaces in such a way as to raise the wetting angle beyond 90° and allow a spontaneous discharge of the gap to take place.

The energy required to expell the liquid from the gap is raised in two steps. According to Volmer's theory, a spherical nucleus is generated in the first step which, however, must contact only the limiting walls and serves the purpose of creating a three phase boundary line. This nucleus and the energy required to form it are much smaller because of the smaller dimensions than the respective values required for the formation of a critical nucleus in the macroscopic theory of nucleation. For the second step (or the following steps) the energy required to expell the liquid is taken mainly from the boundary area energy. This is a completely different physico-chemical process than the process applied as the basis in the direct application of Volmer's theory to the generation of a macroscopical critical nucleus.

This behaviour will be explained on the basis of some examples and their representation in fig. 3 below. The theoretical principles of calculating the energy of nucleation for the two individual steps mentioned and of the nucleation rate are provided in appendices 1 and 2.

Curve O-A-B-C-D in fig. 3 is used to describe the principal sequence of events. As a function of the radius, the energy required to generate a spherical nucleus will increase like a parabola along the curve O-A (appendix 2, equation 13). At point A the spherical nucleus contacts the walls of the parallel gap assumed to have a gap width of 12 \AA . Only at this point a three phase boundary line may be generated between the solid wall, the liquid and the vapour. In this way, at the wetting angle θ of 130° assumed here, wetting energies are released corresponding to the jump from point A to point B in fig. 3. At the same time, this increases the volume of the nucleus as a result of the difference between states A and B from fig. 4.

At point A in fig. 3 the maximum required nucleation energy - achieved by energy fluctuations under Volmer's theory - has been reached. According to equation 11 in appendix 2, the energy of nucleation A_K (which is calculated by equation 13, appendix 2 or equation 1, appendix 1) results in a nucleation rate which can be taken from the scale on the right hand side of the diagram. For point A the nucleation rate thus is approximately $2 \cdot 10^6$ per cm^2 of gap wall area and second.

A further increase in volume as the nucleus grows from point B to C due to the retreat of the liquid requires additional energy in the example chosen. Only after point C the nucleus will be completely stable and continue to grow independently, releasing energy of formation in the direction of D. The transition from point A to C actually is a dynamical process which must not necessarily pass through point B; hence, it may be assumed in the example chosen that the nucleus is stable already after point A. If, energetically speaking, point C should be higher than point A, the energy at point C would have to be raised by the energy fluctuations under Volmer's theory and would determine the rate of nucleation.

Parameters greatly influencing the nucleation rate are the gap width h and the wetting angle θ . For large wetting angles θ

the nucleation rate is determined by the generation of the spherical nucleus corresponding to point A (in fig. 3) and thus by the gap width h . For smaller wetting angles only slightly above 90° point C will be the highest one, energetically speaking, which makes the wetting angle θ determine the nucleation rate. Compared with these influences, for this range of microscopic nucleation superheating of the liquid is of secondary importance only.

In fig. 3 curve O-A shows the nucleation energy calculated by Volmer's theory for spherical nuclei; the curve shows a marked decrease of the frequency of nucleation with increasing radius of the nuclei. For realistic macroscopic radii of nuclei (for instance $1 \mu\text{m}$), which occur in technical boiling processes, the rate of nucleation calculated according to Volmer decreases to unbelievably small values.

The Transition to the Macroscopical Critical Vapour Nucleus

In order to allow the microscopical vapour nuclei generated in the way outlined above to grow to the critical size required for macroscopical nucleation, certain geometrical and physico-chemical conditions must be fulfilled: The channels or gaps in which the process of expulsion takes place must widen in the direction towards the surface of the solid.

The energy required for the increase in volume, which is not raised by the vapour pressure only at this phase of the process, requires the right wetting conditions over the entire way of liquid expulsion, that is to say, the retreat of the liquid must be supported by wetting angles larger than 90° .

The latter condition is explained in fig. 5 in which limiting channel shapes are shown. In these cases, the wetting angle is the parameter. For all shapes of channels having smaller angles of aperture at a given wetting angle ($\theta > 90^\circ$) the vapour nucleus will grow from the microscopic nucleus outlined above into the region of a macroscopical nucleus. If the macroscopical condition has been reached, there is no difference relative to

model concepts based on gas nuclei, as far as the criticality of a nucleus is concerned.

The Statistical Behaviour of Macroscopical Critical Vapour Nuclei

As a consequence of the physico-chemical interface processes, such as corrosion, erosion, diffusion, adsorption, changes of equilibrium parameters etc., the generation of macroscopic void nuclei of the type outlined above cannot be explained statically. This is a dynamical equilibrium between the generation of new and the disappearance of aged void nuclei.

The processes mentioned above continuously change the surface. Existing boiling nuclei can be removed due to erosion of the surface. New nuclei may be generated by the agglomeration of corrosion products or the exposure of new microcracks. The wetting conditions within existing fissure systems are changed by such processes as adsorption, diffusion, chemical reactions so that void nuclei can be activated or deactivated. All these processes are highly dependent both on temperature and the material composition of the entire system.

These influences upon the generation and annihilation of macroscopic void nuclei indicate that this is a dynamical process. The activation of nuclei is influenced by statistics. *)

These statistical effects become particularly pronounced in the numerous experimental studies /14, 15/ of boiling events with liquid alkali metals because it is precisely the liquid alkali metals which have a very high chemical aggressiveness relative to the wall materials. The results of these boiling experiments show that there are major fluctuations in the statistical distribution of the absolute values of liquid superheat, similar to the effects of the so-called period of waiting,

*) The authors realize that this is an unsatisfactory and very general description. However, they would like to indicate that despite obvious progress in physico-chemistry over the past 50 years nobody has as yet succeeded in establishing a more generally useful theory of nucleation.

that is to say, greatly differing periods of time between exceeding the boiling point and the generation of the first vapour bubble /16, 18/.

Conclusion

The hypothesis advanced in this paper is based on physico-chemical processes in its most important elements. In this way, it contains the potential of explaining the statistical effects observed in the experiments and the complete suppression of boiling events in a broad range of states /8/. In our opinion, this constitutes a major advantage over the deterministic models on the basis of gas nuclei used so far.

There is a certain degree of similarity with the model concepts based on gas nuclei of this hypothesis in as much as geometrical conditions and material constants play a part in either model. However,

the very special geometrical conditions and
spatial independent wetting angle

as included in the gas nucleus model have been replaced here by

indeterminate geometrical conditions and a
spatial dependent wetting angle.

In this way, this hypothesis fully takes into account the true physico-chemical and geometrical conditions existing in boiling processes at phase boundaries in heat transfer systems. This immediately gives rise to the conclusions that a theoretical and quantitative treatment of nucleation associated with boiling processes in technical systems is going to meet with extraordinary difficulties.

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Appendix 1

Calculation of the Nucleation Energy of Flat Rotational Symmetrical Nuclei in Parallel Gaps

The energy of nucleation A_k results from the surface formation energy A_o and the expansion energy A_v according to

$$A_k = A_o + A_v \quad (1)$$

Using

- O_l = Liquid surface of the nucleus
- O_s = Solid surface of the nucleus
- $\sigma_{v,l}$ = Surface tension vapour/liquid
- $\sigma_{l,s}$ = Surface tension liquid/solid
- $\sigma_{v,s}$ = Surface tension vapour/solid
- p_v = Pressure of vapour in the nucleus
- p_l = Pressure in the liquid
- V = Vapour volume of nucleus

we obtain

$$A_o = \sigma_{v,l} \cdot O_l - (\sigma_{l,s} - \sigma_{v,s}) \cdot O_s \quad (2)$$

$$A_v = -(p_v - p_l) \cdot V \quad (3)$$

The vapour pressure p_v over curved liquid surfaces of the radius of curvature r_o results from

$$p_v = p_\infty \cdot \exp \left(- \frac{2 \cdot \sigma_{v,l}}{r_o} \cdot \frac{\bar{V}_l}{RT} \right) \quad (4)$$

where

- p_∞ = Vapour pressure over the plane surface
- \bar{V}_l = Molar volume of liquid
- R = Gas constant
- T = Absolute temperature

For the wetting angle θ between the liquid and the wetted solid the following relationship applies:

$$\cos \theta = \frac{\sigma_{v,s} - \sigma_{l,s}}{\sigma_{v,l}} \quad (5)$$

For a rotational symmetrical nucleus in a parallel gap corresponding to fig. 6 the necessary quantities for calculation of the nucleation energy result from the following relations:

$$Q_s = 2 \cdot \pi \cdot \left[r - \frac{h \cdot (1 - \sin \theta)}{2 \cdot \cos \theta} \right]^2 \quad (6)$$

$$Q_l = \frac{\pi \cdot h}{\cos \theta} \cdot \left[r \cdot (\pi - 2\theta) + h \cdot \left(1 - \frac{\pi - 2\theta}{2 \cdot \cos \theta}\right) \right] \quad (7)$$

$$V = 2 \cdot \pi \cdot \left[(r - l) \cdot h + F \right] \cdot R_F \quad (8)$$

with

$$l = \frac{h \cdot (1 - \sin \theta)}{2 \cdot \cos \theta} \quad (8.1)$$

$$F = \frac{h^2}{8 \cdot \cos^2 \theta} \cdot \left[\pi - 2\theta - \sin(\pi - 2\theta) \right] \quad (8.2)$$

$$R_F = \frac{r - l}{2} + \left[\frac{r + l}{2} - \frac{h}{2 \cdot \cos \theta} + \frac{h^3}{12 \cdot F} \right] \cdot \frac{F}{F + h \cdot (r - l)} \quad (8.3)$$

The mean radius of curvature r_0 of the liquid surface is approximately represented by the vertex value, thus

$$r_0 = 2 / \left(\frac{1}{r} + \frac{2 \cos \theta}{h} \right) \quad (9)$$

If the molar volume of the vapour is large compared with the molar volume of the liquid (which will be true in most cases), the vapour pressure p_∞ is taken in a good approximation from the vapour pressure curve of the liquid for the superheat temperature considered. Hence, from equations (3) and (4) one obtains explicitly

$$A_V = - \int p \cdot \exp \left(- \frac{2 \sigma_{V,l}}{r_0} \cdot \frac{V_l}{R \cdot T} \right) - p_l \cdot V \quad (10)$$

which allows equation (1) to be evaluated.

Appendix 2

Calculation of the Nucleation Probability for Spherical Nuclei in Parallel Gaps

The nucleation probability I in a system with N molecules according to M. Volmer /1/ turns out to be

$$I = N \cdot \sqrt{\frac{6 \cdot \sigma_{V,l}}{\pi \cdot m \cdot \left(2 + \frac{p_l}{p_\infty}\right)}} \cdot \exp \left(- \frac{H + A_k}{kT} \right) \quad (11)$$

where

m = Mass of the molecule

H = Molecular heat of evaporation

k = Boltzmann constant

A_k = Energy of formation of the nucleus

For the other terms, see appendix 1.

For a critical spherical nucleus it holds that

$$A_k = \frac{16 \cdot \pi \cdot \sigma_{V,l}^3}{3 \cdot (p_V - p_l)^2} \quad (12)$$

whereas for a spherical nucleus of any size

$$A_k = 4 \cdot \pi \cdot \sigma_{V,l} \cdot r^2 - \frac{4 \cdot \pi \cdot (p_V - p_l) \cdot r^3}{3} \quad (13)$$

Here r is the radius of the sphere. For p_V , again equation (4) (appendix 1) can be taken.

If the number of molecules N (= number of liquid molecules in the parallel gap) is referred to the unit wall area of the parallel gap, using $r = \frac{h}{2}$, equations (11) and (13) may be used to calculate the frequency of formation of spherical nuclei of diameter h referred to unit area and unit time. Hence,

$$N = \frac{h \cdot N_L}{\bar{V}_l}, \quad (14)$$

where

N_L = Loschmidt number

h = Width of the gap

\bar{V}_l = Molar volume of liquid.

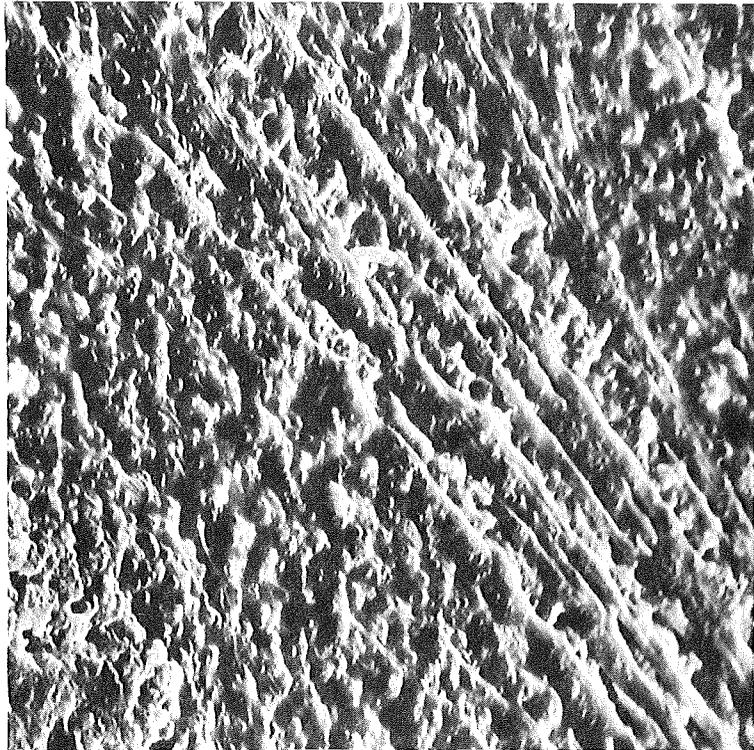


Fig. 1 Stainless Steel Surface, Material Number 4981, Filed
(Electron Scanning Microscopy, Enlargement 2350:1) /10/

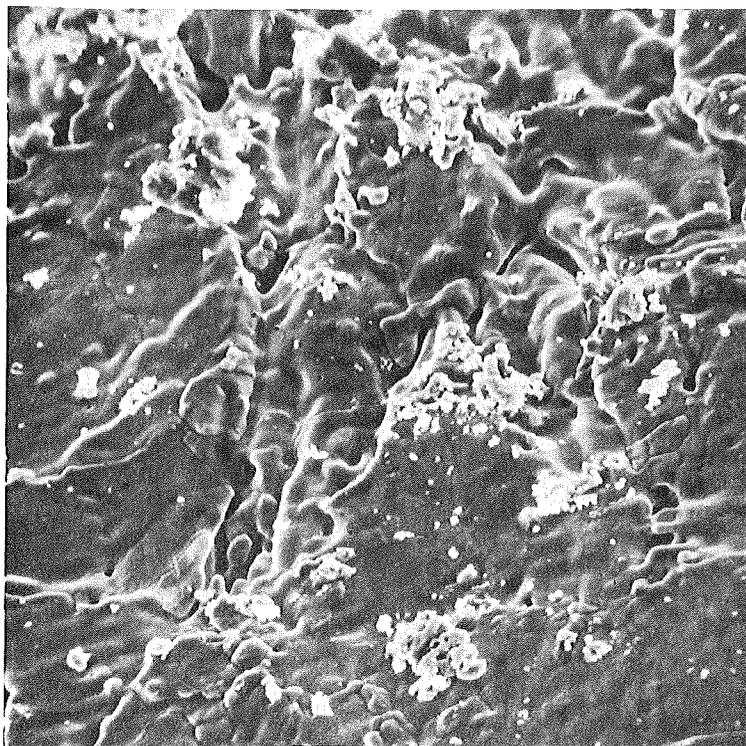


Fig. 2 Nickel Surface (Inner Tube Wall) Drawn after Insertion
into a Sodium Loop
(Electron Scanning Microscopy, Enlargement 1050:1) /10/

Energy of Nucleation

[10^{-12} erg]

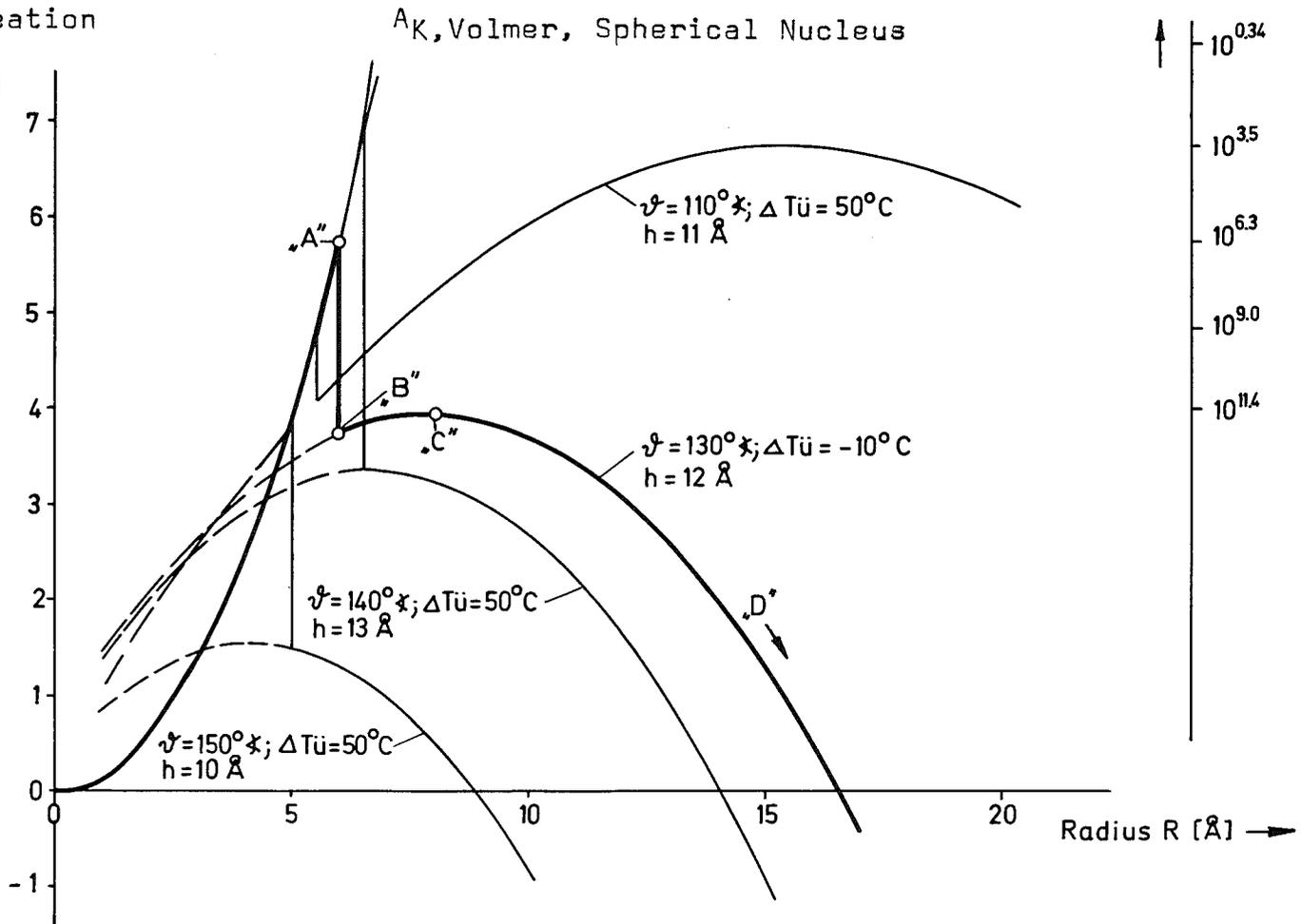
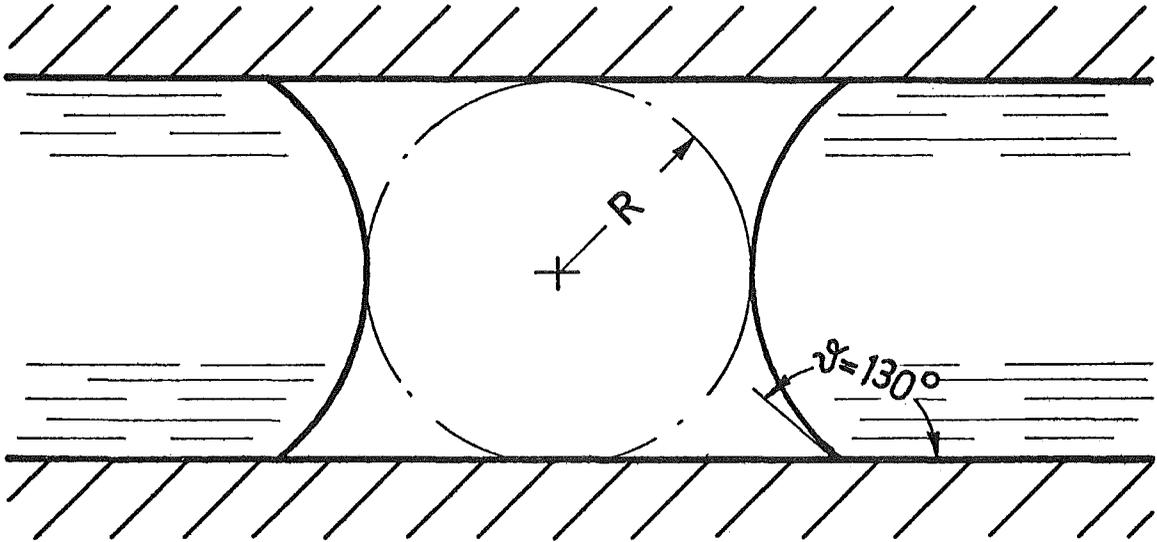
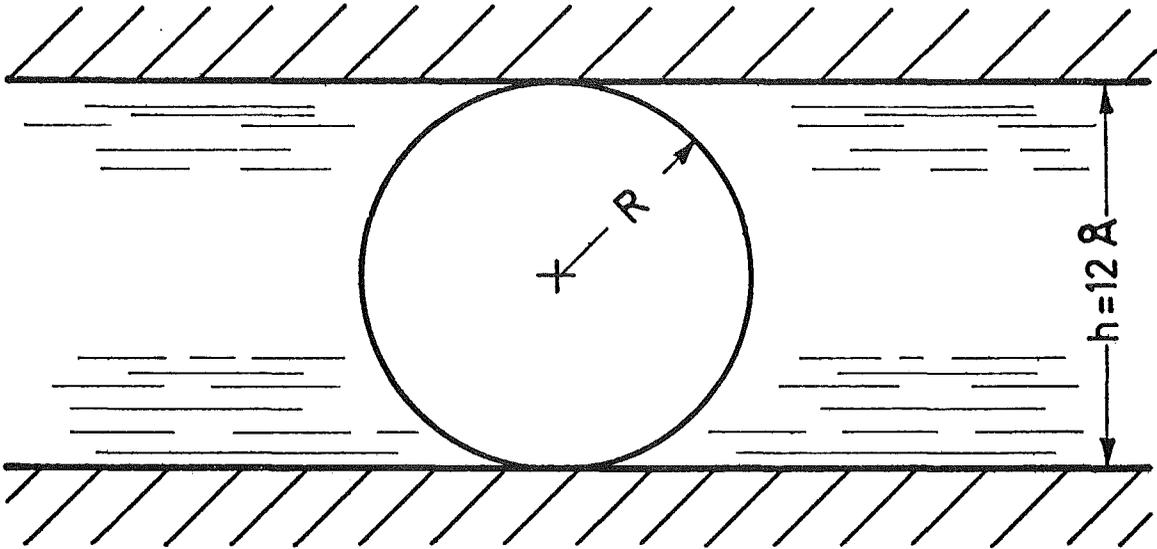


Fig. 3

Examples of the curve of nucleation work calculated for narrow gaps as function of the parameters radius R , gap width h , wetting angle θ and superheat $\Delta T_{\text{ü}}$ (cf. appendices 1 and 2)

State "A"



State "B"

Fig. 5 Transition from a spherical nucleus to a nucleus with three phase boundary lines

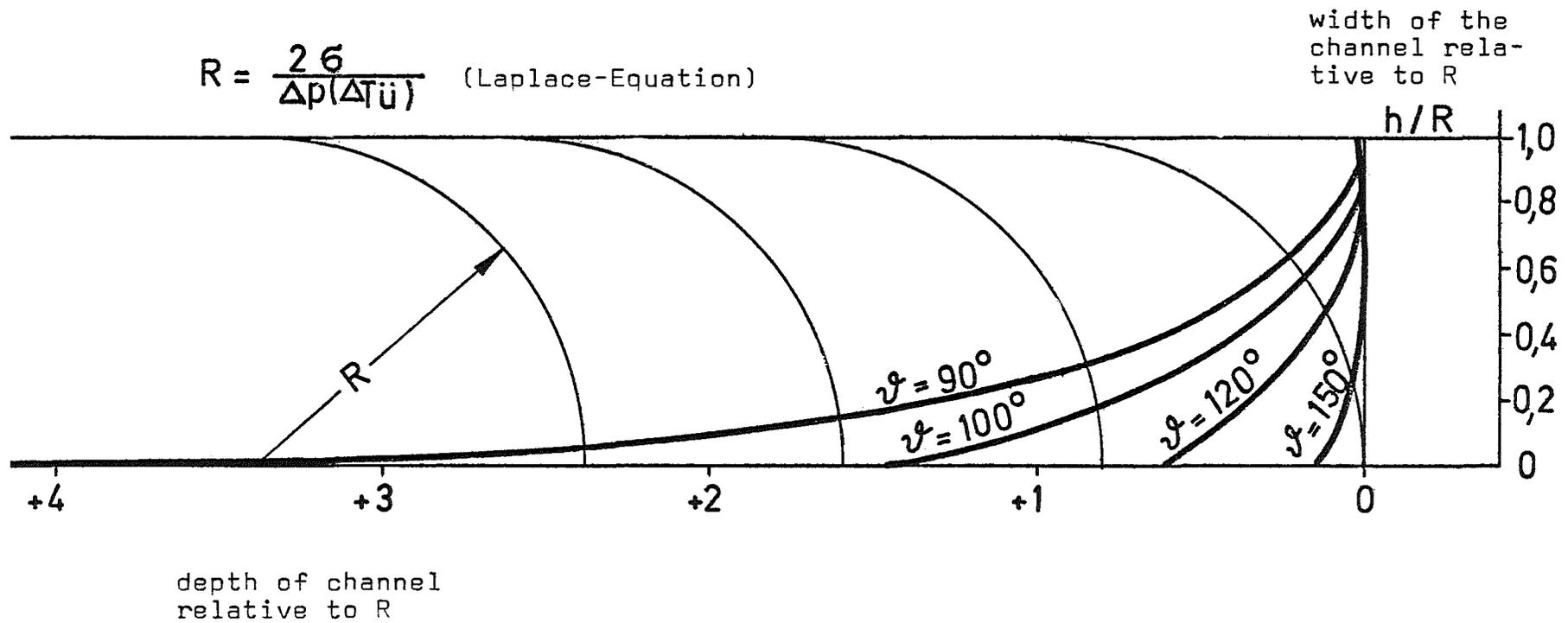


Fig. 5 Limiting channel shapes, for gaps or rotational symmetrical apertures at constant radius R of the nucleus (or constant superheat $\Delta T_{\bar{u}}$) as function of the wetting angle θ

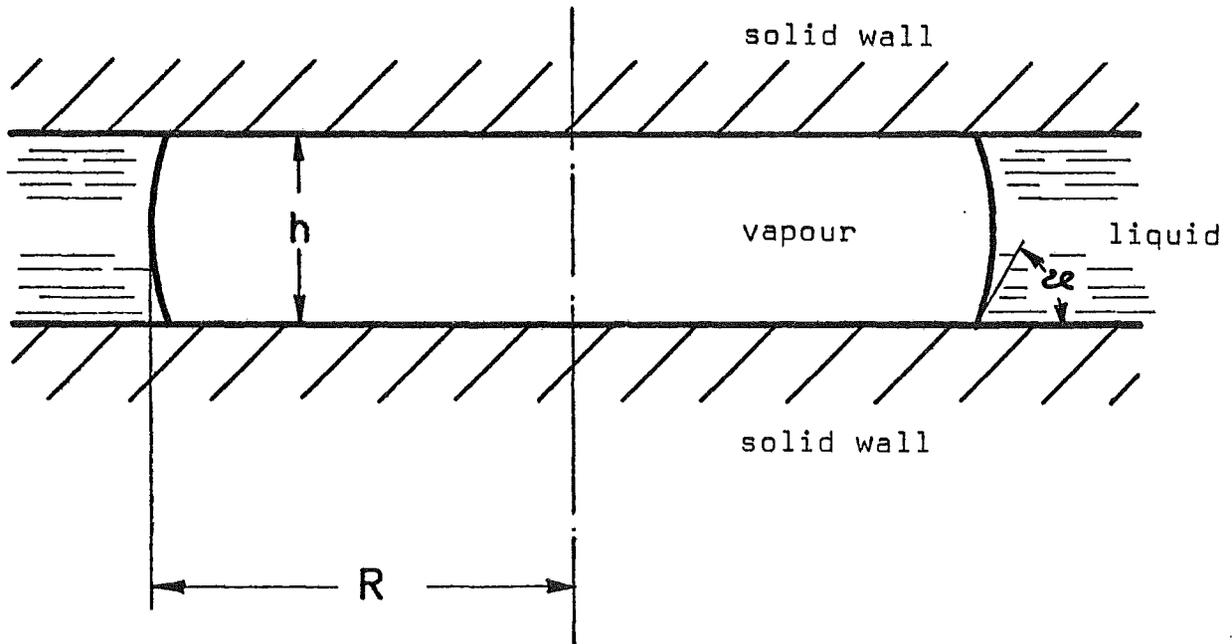


Fig. 6 Geometry of a rotational symmetrical nucleus in a parallel gap