

## **POOL BOILING IN HIGHLY VISCOUS MIXTURES – DESIGN OF A NEW APPARATUS AND RESULTS OF PRETESTS**

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### **Abstract**

In a project which deals with degassing of polymer solutions using pipe-bundle heat exchangers, noticeable discrepancies have been observed between experimental results for boiling highly viscous mixtures and predictions of available correlations. These differences revealed shortcomings in understanding and modeling of the process. An experimental project is presented, which focuses on fundamentals of heat and mass transfer in boiling highly viscous mixtures. Effects that have been predicted for boiling in such mixtures are discussed.

In order to distinguish between the influence of viscosity and marginal vapor pressure of the highly viscous component, an extremely wide-boiling model system with low viscosity will be investigated. A standard apparatus for measuring heat transfer in an equilibrated pool is modified for measurements on such systems. The design of the modified apparatus is described. Additional modifications necessary to adapt a standard apparatus to wide-boiling highly-viscous mixtures are briefly discussed.

Results are presented for mixtures of n-pentane and poly dimethyl siloxane (silicon oil) with a viscosity of  $10^6$  m Pa s boiling in a simple pod-boiling equipment at ambient pressure. Temperature distributions in the pool are reported and nucleation mechanisms are discussed. A new mechanism is observed for bubble formation above the test tube.

### **1. Introduction**

As part of an industrial project with the Bayer Technology Services GmbH, flow boiling heat transfer in highly viscous mixtures is investigated in an annular gap and a down pipe model system. Wienecke *et al.* (2005) reported details on the test facility. The objective of this project is to model and optimize the process for industrial applications. Attempts to model the process with established correlations for flow boiling revealed large deviations between calculated and measured heat transfer coefficients, even though the correlations were adapted and extended by viscosity dependent terms. Large deviations are particularly observed for the transition from subcooled to nucleate boiling and for convective two-phase boiling. These strong discrepancies indicate fundamental problems in characterizing heat and mass transfer in highly viscous mixtures.

In the last few years an increasing number of investigations has been concerned with heat transfer in boiling mixtures. However, most of the studies are limited to mixtures with low viscosity, such as organic liquids and aqueous solutions, and refrigerant-oil mixtures; for an overview see, e.g., Gorenflo and Köster (1999). In a certain way, heat transfer in highly viscous mixtures is similar to refrigerant-oil mixtures, which consist of one component with low and one with high viscosity forming a wide boiling mixture. Mohrlök *et al.* (1998) give a review of experimental investigations and empirical and semi-empirical correlations describing the heat transfer in boiling refrigerant-oil mixtures. Most of the investigations initially showed an improvement in heat transfer in mixtures with small oil fractions; when increasing the oil fraction heat transfer greatly deteriorates. Diffusion

of the refrigerant is inhibited by concentrated oil at the heated surface or the phase interface. Due to a reduced bubble growth rate, the wall temperature of the heated surface increases. In most cases, investigations are limited to oil fractions below 10 %. The results of these experiments and models are not directly applicable to systems containing a large fraction of the highly viscous component.

Plastics industry has also conducted research of boiling highly viscous mixtures. The few published experimental and theoretical attempts are predominantly targeted at describing polymer degassing processes for engineering purposes. In most studies the objective is the design and optimization of extruders, falling film or slit processes and not a fundamental understanding of the processes. Astarita and Maffettone (1994) give a critical review of existing models for characterizing heat, mass and momentum transfer. According to them, nucleate boiling is generally limited to bubble growth in highly viscous mixtures. The model of Amon and Denson (1984) describes bubble growth depending on viscosity and diffusion and forms the basis for subsequent models. The model of Maffettone and co-workers (see Maffettone *et al.* (1991) and Ianniruberto *et al.* (1993)) includes heat, mass and momentum transfer to describe polymer degassing in a single and multi slit, which can be compared to degassing in a pipe. By using this model, pressure and temperature profiles as well as volatile concentration at the slit end can be calculated. Comparisons of calculated and experimental results show deviations of approximately 10 %.

Until now, fundamental questions like nucleation, bubble collaboration and coalescence have not been satisfactorily answered. Astarita and Maffettone (1994) state that heat transfer plays an important role in degassing processes; nevertheless, it is neglected in numerous models. Influences of physical properties, such as viscosity, thermal conductivity and vapor pressure have not been sufficiently investigated. Theoretical investigations are interpreted in various ways due to the scarcity of experimental data.

Gerber and Span (2005) presented a concept for an experimental study, which focuses on the fundamental understanding of processes with boiling highly viscous mixtures. This paper reports first results of this ongoing project. In parallel, Liesenfelder *et al.* (2006) present the status of the technically focused flow-boiling experiments and first attempts to model the corresponding processes. For reasons explained by Wienecke (2006) and Gerber and Span (2005), mixtures of n-pentane and silicone oil with nominal viscosities ranging from  $10^4$  to  $10^6$  m Pa s are used as model system for boiling highly viscous mixtures in both projects. This project additionally involves mixtures of n-pentane and n-hexadecane as a wide boiling reference system with low viscosity.

To introduce into the structure of the project, its objectives, as discussed by Gerber and Span (2005), are briefly summarized in the following section. In Section 3 the design of the apparatus, which is under construction for measuring heat transfer coefficients in the low-viscosity system n-pentane / n-hexadecane, will be described. In Section 4 first results from boiling experiments with n-pentane / silicon-oil mixtures in a subcooled pool will be shown.

## 2. Objectives

For engineering purposes, the characterization of boiling processes traditionally focuses primarily on the quantification of the heat transfer coefficient or the wall superheat in dependence on heat flux, pressure, concentration and, where required, viscosity. Empiric and semi-empiric correlations have been developed for pure fluids and mixtures with low viscosity to describe these relationships with accuracy adequate for technical use. One objective of this project is to establish a database, which will allow for a semi-empiric description of boiling highly viscous mixtures later on. However, preliminary studies and experience with practical applications show that some fundamental effects have to be considered when dealing with boiling of highly viscous mixtures. These effects play a minor role or are completely irrelevant for fluids with low viscosity. In many

cases, the influence of high viscosity on the pool boiling process can be derived from the knowledge of the underlying physical effects.

In this section a number of phenomena will be discussed, which were observed or are expected for boiling of highly viscous mixtures. Prior to a systematic quantitative characterization of heat transfer, the influence of these phenomena needs to be understood. Some of them may simply be considered parameters of the used apparatus, others are of fundamental physical importance.

*Transition from pool boiling to flow boiling:* The incident flow effects, which can be found in all standard facilities, have proven to be irrelevant for boiling fluids with low viscosity. At low flow rates the heat transfer caused by bubble formation and the resulting natural convection is predominant. In highly viscous fluids, the development of natural convection is reduced to a minimum; thus, the incident flow caused by unavoidable forced revolution may have a much stronger effect on heat transfer. It is crucial to determine a critical flow rate, up to which this effect can be disregarded - not only for subsequent experimental and theoretical studies, but also for technical applicability.

*Definition of a reference temperature:* In boiling mixtures with low viscosity, it is assumed that a constant composition is reached within close proximity of the heated surface; thus, the saturation temperature becomes a constant that is used as reference, e.g., for calculation of heat transfer coefficients. However, highly viscous mixtures will develop a larger concentration gradient in the pool. Consequently, the definition of the superheat relevant to heat transfer may have to be modified. The temperature distribution in the pool has to be measured by an array of thermocouples or by moveable sensors and a suitable reference temperature has to be defined.

*Use of reduced pressures:* In characterizing the pressure dependence of boiling in pure fluids and mixtures, the use of the reduced pressure is an established method. Usually, a critical pressure - the reference for the reduced pressure - cannot be determined for highly viscous mixtures. Since the pressure dependence of heat transfer in boiling fluids is mainly related to characteristics of the vapor phase, the critical pressure of only the pure volatile component can possibly be used to reduce measured pressures. Whether this approach is suitable to describe the pressure dependence of heat transfer in boiling highly viscous mixtures has to be proven.

*Heat and mass transfer into the rising bubble:* Investigations on boiling fluids with low viscosity revealed that heat and mass transfer into the rising bubble is an important contribution to the total heat and mass transfer. Due to the superheat of the surrounding fluid, the fluid or in case of extremely wide-boiling mixtures the volatile component constantly evaporates into the rising bubble. As a result, heat and mass transfer are larger than expected from nucleation and bubble growth at the heated wall. Depending on the concentration of the highly viscous component, a stronger resistance to mass transfer into the bubble is expected due to low diffusion coefficients and thick boundary layers. To verify this thesis, heat and mass transfer into the rising bubble have to be measured by observation of bubble growth.

*Influence of the surface:* The relation between surface structure and heat transfer in boiling fluids with low viscosity has been investigated for a long time. New methods for characterizing technical surfaces led to a better understanding of the corresponding effects, see for example Luke (2006). Heat transfer in mixtures with high viscosity is strongly affected by convective and diffusion processes - in any case high superheats are generated at the heated wall. It is not clear whether the influence of the surface, which is observed in boiling mixtures with low viscosity, is nonexistent, smaller than or similar in highly viscous mixtures. If a significant influence of the surface is observed, new or yet unconsidered approaches for optimizing technical processes become possible.

*Critical heat flux of highly viscous mixtures:* Increasing viscosity of the liquid phase inhibits the flow of vapor bubbles. Due to an accumulation of vapor on the heated surface, film boiling may occur at much lower heat fluxes than in mixtures with low viscosity. Until now, the dependence of critical heat flux on viscosity of the liquid phase has not been investigated systematically.

At high viscosities and low concentration of volatile component Gerber and Span (2005) predicted the formation of a liquid film on the heated wall, which is almost completely depleted of the volatile component. Bubble nucleation would be moved from the heated wall to the superheated liquid. In this case, high superheats arise due to insulation by the non-evaporating component. Experimental evidence for this effect is given in Section 4.

*Temperature gradients:* In order to determine the heat transfer coefficient, the wall temperature and the pool temperature need to be obtained. The temperature of the heated wall can be measured by thermocouples soldered into the tube wall. This allows for an investigation of the mean superheat as well as for a radial resolution of the superheat over the tube perimeter.

The measurement of the temperature distribution inside the pool will be accomplished by an array of thermocouples arranged in the pool above and below the heated test tube. Since a temperature gradient is assumed from the test tube towards the surface and towards the bottom of the pool, the thermocouples are also used to localize a suitable reference temperature in the liquid phase.

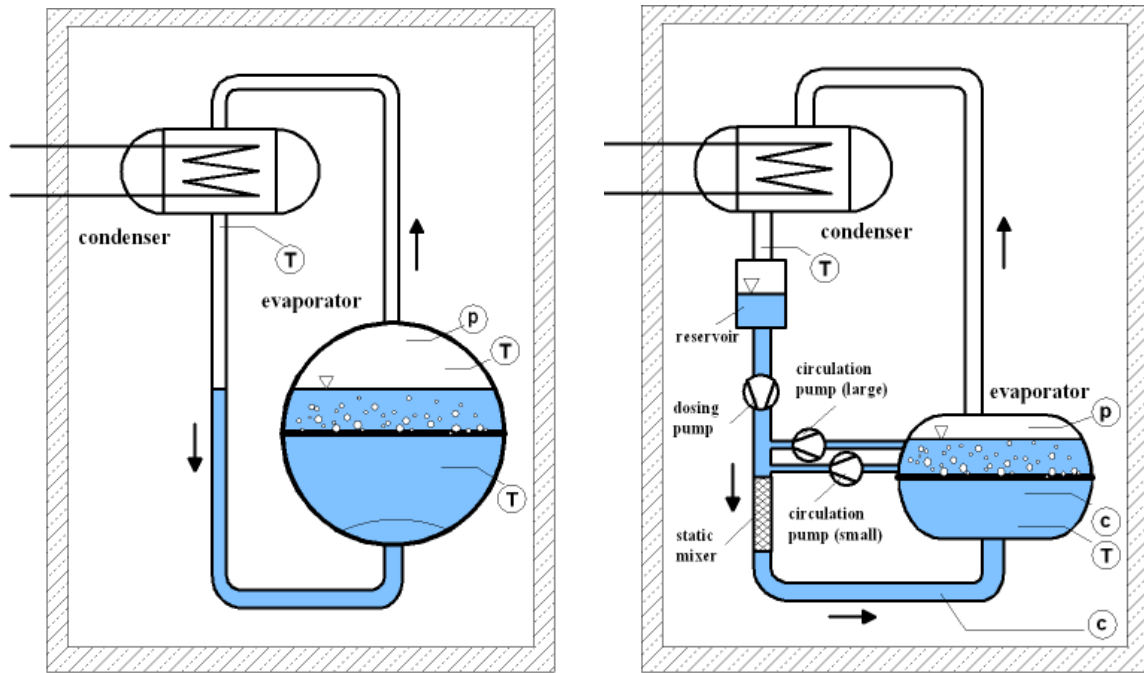
*Composition gradients:* When dealing with mixtures with low viscosity, a constant composition is assumed for the pool outside of a boundary layer due to intensive mixing of the pool. In highly viscous mixtures mixing is less intensive and composition will differ significantly across the pool. The composition of the mixture supplied to the pool can be measured online by a process refractometer. A refractometer that can be integrated into the pool will soon become available. Used with sensors in different positions, this refractometer will supply some information on composition gradients in the pool. Raman spectroscopy is considered a means to measure composition gradients with higher spatial resolution, see Gerber and Span (2005). However, this technology has not been tested in pools with boiling highly viscous mixtures yet.

*Influence of viscosity versus influence of asymmetric phase-equilibrium:* Like the technically relevant systems, the highly viscous n-pentane / silicon oil mixtures considered here are extremely wide boiling; the concentration of silicon oil in the formed vapor-phase is negligible. Effects caused by high viscosity have to be separated from effects caused by this very asymmetric phase equilibrium. Experimental data on heat transfer in boiling mixtures with similar phase equilibrium conditions are hardly available. The available empirical correlations for the resulting heat transfer coefficient seem to be not applicable to the kind of systems considered here, see Span *et al.* (2005). Therefore, heat transfer in boiling mixtures of n-pentane and n-hexadecane will be investigated experimentally to establish a reference for a comparable wide boiling system with low viscosity.

Silicon oil is commercially available with nominal viscosities ranging from  $10^3$  to  $2 \cdot 10^6$  mPa s. Beside the large difference in viscosity most other thermodynamic properties of the silicon oils are hardly different. In this way the influence of viscosity on pool-boiling heat-transfer can be studied in a wide range of viscosities. However, surface tension may vary significantly with viscosity. Whether this effect is relevant and how it can be separated from the effects caused by viscosity needs to be investigated.

### **3. Layout of the Apparatus**

In order to investigate the given model systems, the standard apparatus by Götz (1980) needs to be modified. Figure 1 shows the basic layout of the conventional and the modified apparatus. The

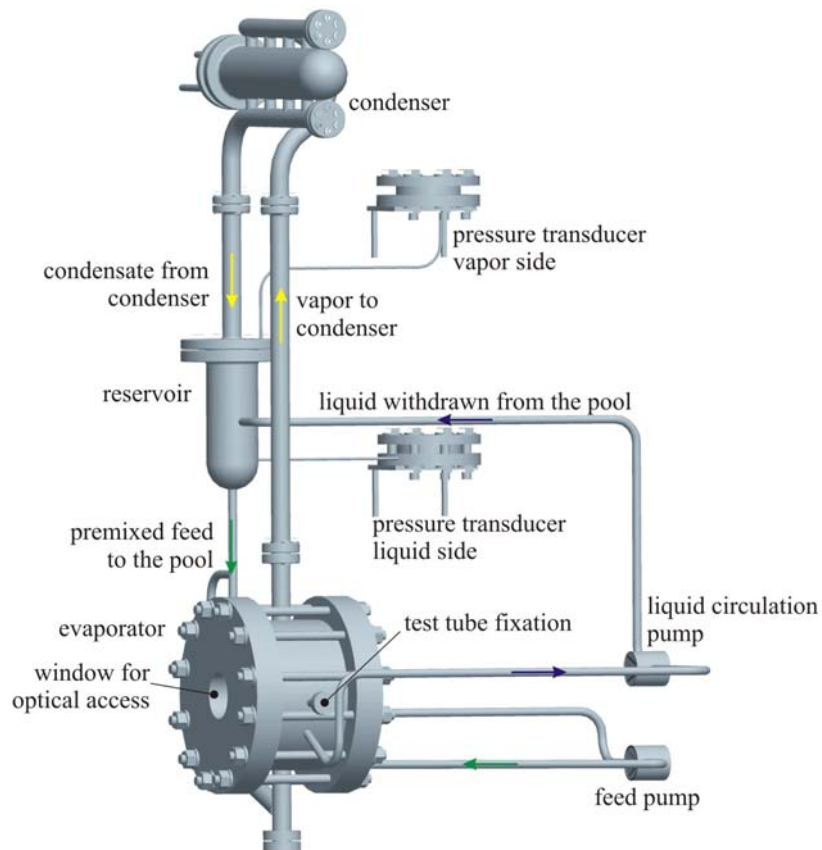


**Figure 1:** Basic layout of the standard apparatus by Götze (1980) and the modified apparatus proposed by Gerber and Span (2005). p – pressure measurement, T – temperature measurement, c – composition measurement.

conventional standard apparatus uses the principle of natural convection. Vapor phase is constantly formed by boiling. In the condenser vapor phase is condensed and led back, forced by gravity, into the pool. Due to natural convection in the pool created by bubble formation and rise, an adequate mixing of the components is expected.

When working on mixtures, the phase interface separates liquid and vapor with different composition. The composition of the pool needs to be monitored if the amount of fluid in the vapor phase, in the condenser and in the connecting tubes is not negligible. The condensate, which contains an increased concentration of the low boiling component, needs to be cooled to a temperature below the equilibrium temperature of the pool. In wide boiling mixtures this temperature difference becomes too large to enable a remixing of the liquid in the pool with the condensate without causing significant temperature gradients in the pool or bubble formation in the feed. Natural convection has to be replaced by forced convection with premixing of condensate and liquid withdrawn from the pool. The temperature of the premixed fluid has to be increased to almost equilibrium temperature before it is supplied to the pool.

Figure 2 shows a more detailed drawing of the apparatus currently under construction for investigation of wide boiling mixtures with low viscosity; Figure 3 shows a photo of the open evaporator with a mounted test tube. Forced convection is realised by combination of a feed pump and a circulation pump withdrawing liquid from the pool. The mixing ratio between condensate and circulated liquid is controlled by adjusting the volume flow of the circulation pump. The liquid level in the reservoir, where condensate and circulated liquid are premixed, is measured using a differential pressure indicator connected to the system via pressure transducers. To control the liquid level, the volume flow in the feed pump is controlled. The evaporator allows for optical access via a window and for fixation of test tubes with different diameter.



**Figure 2:** Layout of the modified apparatus that is currently under construction for investigation of pool boiling heat transfer in wide boiling mixtures.



**Figure 3:** Photo showing the open evaporator with a mounted test tube. The thermocouples installed in the test tube allow for wall temperature measurements with high peripheral resolution. The premixed feed enters the pool at the bottom. Vapor leaves the evaporator at the top. Liquid for the circulation loop is withdrawn from a level above the test tube through the tube installed on the right side of the evaporator.

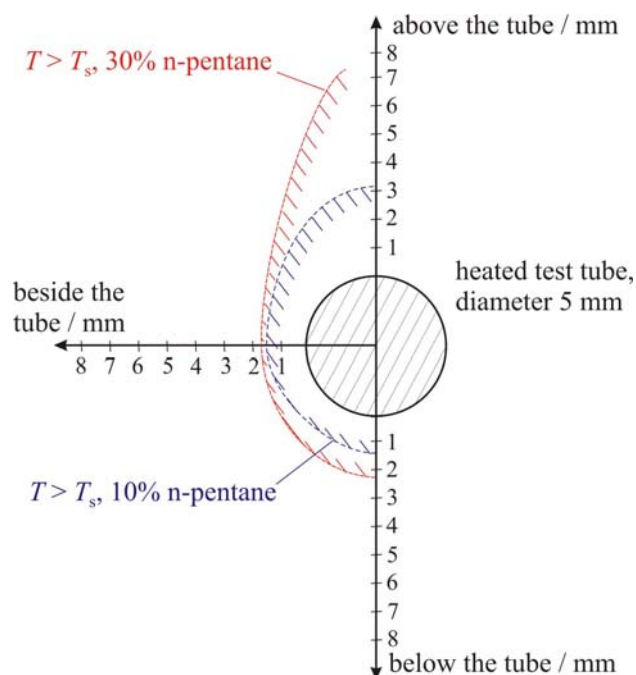
A second standard apparatus will be modified for experiments with highly viscous mixtures. To be able to work with these systems, a number of constructional details have to be adapted. For example, tube diameters in the circulation and feed loop have to be increased. A static mixer has to be integrated and the locations of the pumps have to be modified as shown in Figure 1 to ensure proper flow. Process refractometers have to be integrated into the pool. In this way control of the condensate level in the reservoir will allow for a change in the average composition of the liquid in the pool. Beside such adaptations the design of the modified standard apparatus for investigation of pool boiling of highly viscous mixtures will be based on experience gathered with the apparatus described above.

#### 4. Results of Pretests on Boiling Highly Viscous Mixtures

Yet no results are available for boiling highly-viscous mixtures in a properly equilibrated pool; the required modified apparatus is still under construction, see Section 3. However, to gather further experience with this kind of systems a number of experiments were carried out in open subcooled pools at ambient pressure. Results of these experiments are reported in this section.

An electrically heated test tube with an outer diameter of 5 mm and a length of 41 mm was positioned in an open pool at ambient pressure, which was filled with a subcooled mixture of n-pentane and silicon oil with a nominal viscosity of  $10^6$  mPa s. Temperatures in the pool were measured with thermocouples mounted on an adjustable support to be able to measure temperatures in well defined but variable distances to the heated surface. The electrical heater was controlled to a constant power corresponding to a heat flux of  $\dot{q} = 6.06$  kW/m<sup>2</sup>, where stable boiling with conditions favorable for optical monitoring was observed in pretests. A conventional video system was used to monitor the boiling process. Two compositions with mass fractions of 10% and 30% n-pentane were chosen for the experiments. The mixtures were prepared gravimetrically.

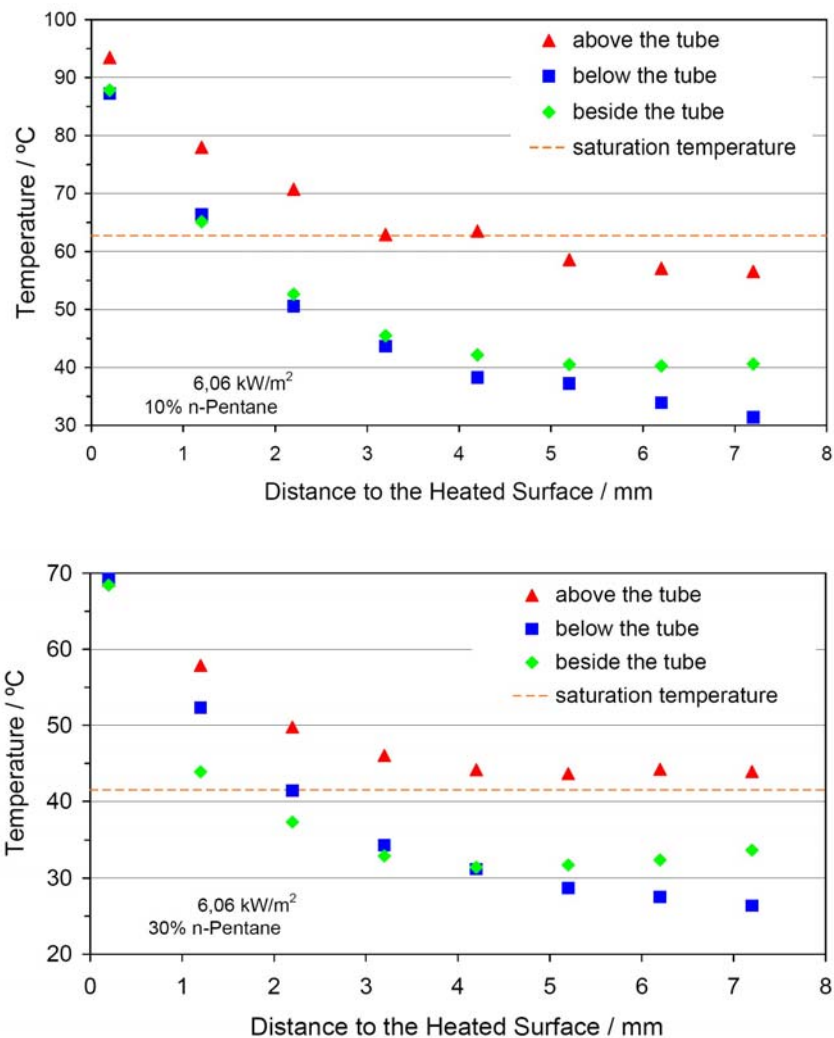
Temperatures in the pool were measured above, beside and under the test tube. Data for locations with a nominal distance of 0.2 mm to 7.2 mm to the heated surface were evaluated. Figure 4 gives



**Figure 4:** Positions where temperatures were measured in the pool in relation to the size of the test tube. The hatched areas indicate regions where temperatures are assumed to be higher than the saturation temperature calculated with average pool composition.

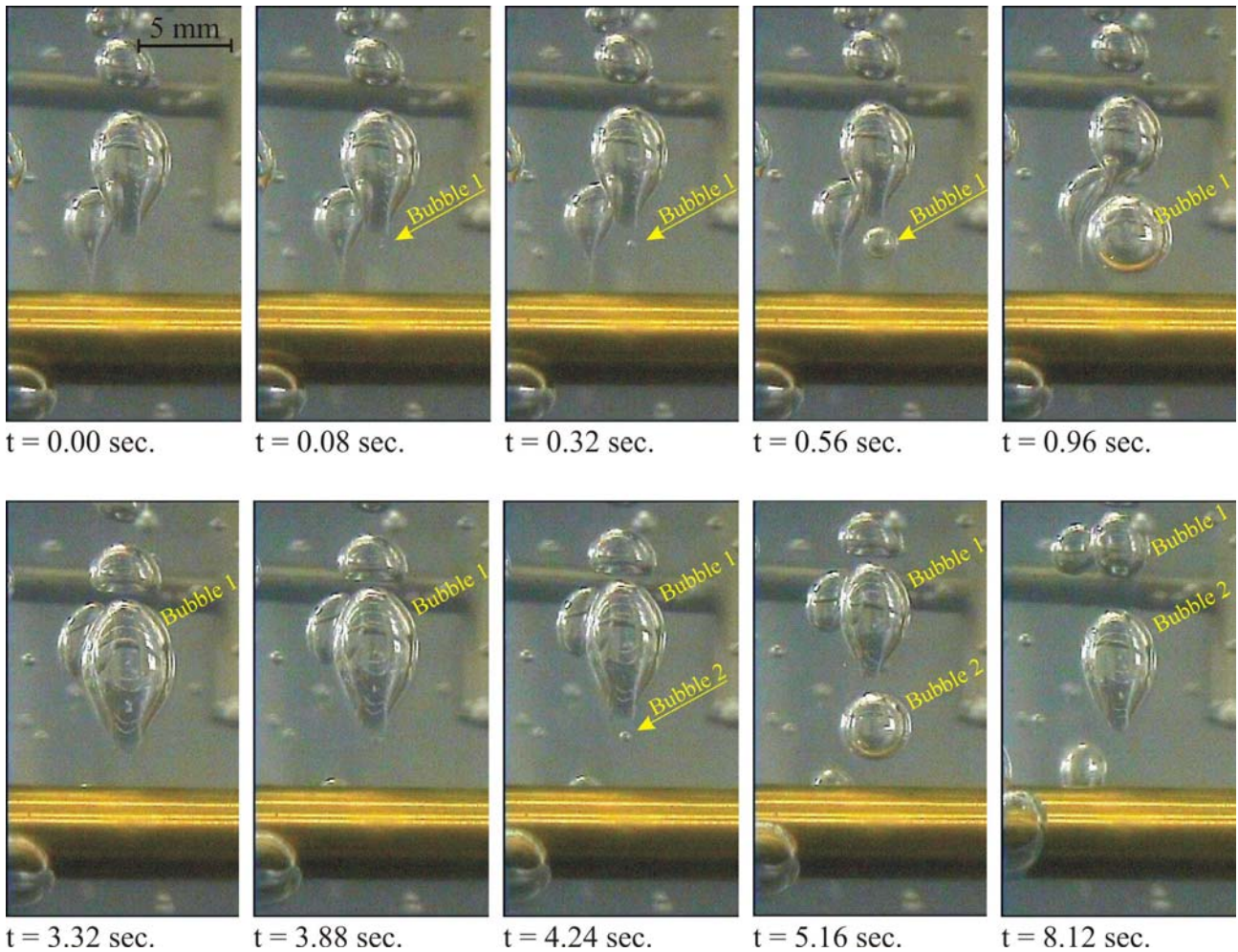
an impression of the positions where temperatures were measured in relation to the size of the test tube. The size of the thermocouple ( $\varnothing 0.5$  mm) limits the spatial resolution of measured temperatures. Steep gradients very close to the heated surface cannot be recorded with this technology.

Figure 5 shows measured temperature distributions for experiments with 10% and 30% mass fraction of n-pentane. Saturation temperatures resulting from calculations for ambient pressure and average pool composition are  $T_{s,10\%} = 62.8^\circ\text{C}$  and  $T_{s,30\%} = 41.5^\circ\text{C}$ . In the mixture with a mass fraction of 10% n-pentane temperatures fall below saturation temperature under and beside the tube within less than 2 mm and above the tube within 3 to 4 mm, see also Figure 4. However, strong temperature gradients were recorded. Close to the tube measured temperatures exceed the calculated saturation temperature by up to 30 K, nucleate boiling is observed. Beginning condensation of the formed bubbles is observed within the measured range of 7 mm above the tube. However, condensation of the bubbles is slow even in areas where the temperature clearly falls below saturation temperature. This effect may be attributed to the fact that the bubbles contain almost pure n-pentane and that diffusion processes, which are required to force the n-pentane back



**Figure 5:** Temperature profiles measured in the pool above, beside and under the test tube. The upper figure shows results for a mixture with 10% n-pentane and the lower figure for 30% n-pentane by mass. Plotted saturation temperatures are calculated with the average composition in the pool and for ambient pressure.





**Figure 6:** Formation of bubbles in the superheated liquid layer above the test tube. Bubble 1 is formed about 2.5 mm above the test tube from a nucleus not visible in this resolution. Bubble 2 is formed at the same location from a residual of bubble 1 4.16 seconds after bubble 1 was formed.

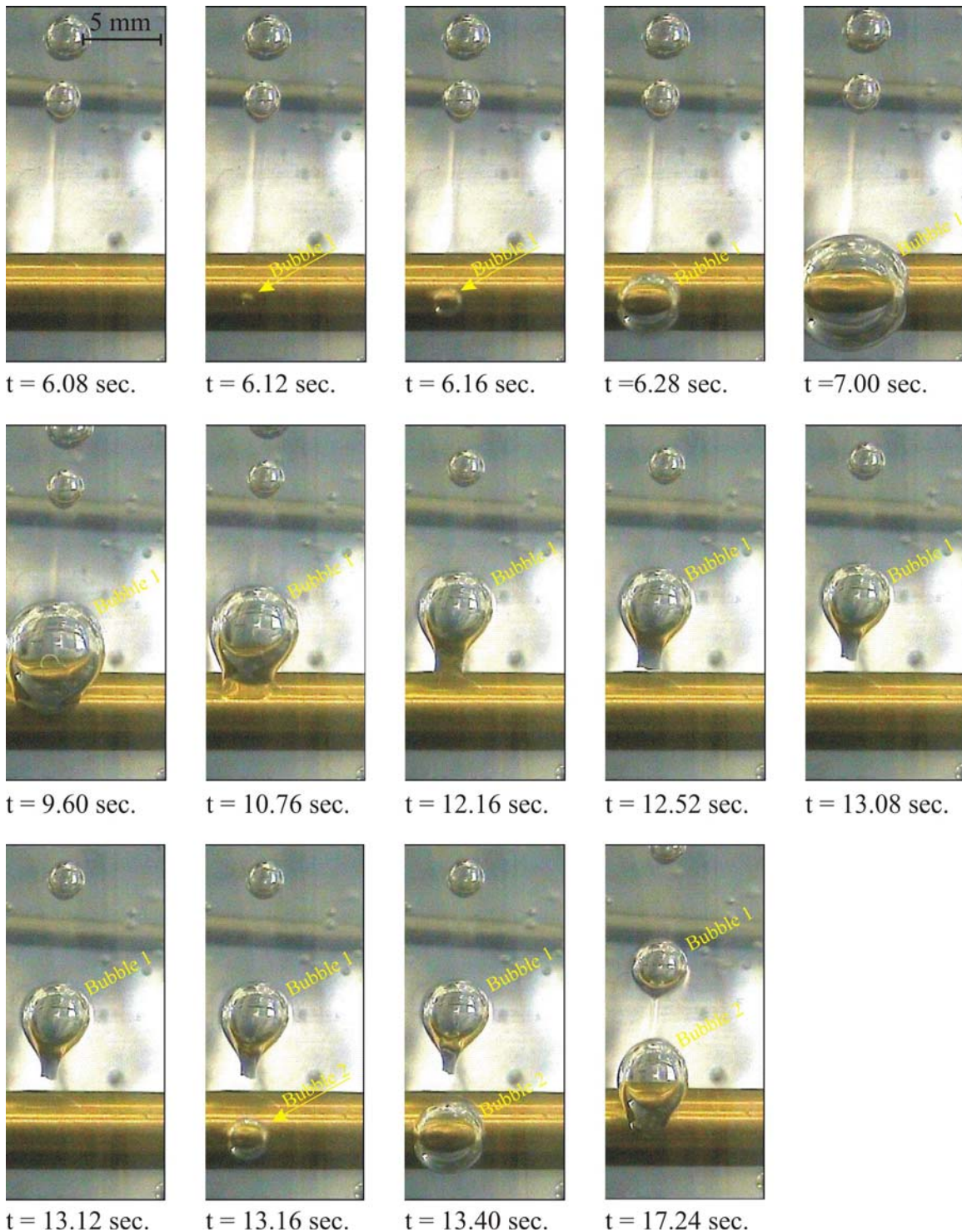
into the liquid mixture, are slow. The saturation temperature of pure n-pentane at ambient pressure is  $T_{s,100\%} = 35.7^\circ \text{C}$ ; direct condensation of n-pentane in the bubbles cannot occur at the measured temperatures.

For the mixture with 30% n-pentane by mass, temperatures fall below saturation temperature within about 2 mm beside the tube as well. However, above the tube measured temperatures remain above the saturation temperature calculated with the average composition of the liquid in the pool for more than 7 mm. Slow condensation of bubbles is observed outside of this range. Bubbles formed at the side of the tube get into contact with subcooled liquid earlier. Both effects can be verified by video recordings. At least for bubbles formed in the superheated liquid layer above the test tube, the results of this experiment are expected to be similar to results to be found in a properly equilibrated pool later on. The average value of the temperature of the heated tube surface is  $T_{\text{tube},30\%} = 88.9^\circ \text{C}$  ( $\pm 1.2^\circ \text{C}$ , determined in three subsequent test runs). The resulting heat transfer coefficient calculated with the saturation temperature at average pool composition is  $\alpha_{30\%} \approx 128 \text{ W}/(\text{m}^2 \text{K})$ .

Figure 6 shows the formation of bubbles in the superheated liquid layer above the test tube. At the time  $t = 0.08 \text{ sec.}$  a nucleus becomes visible in the wake of a preceding bubble about 2.5 mm above the tube, which grows to a bubble with a diameter of about 4.5 mm within less than one second. During this process the distance between bubble and surface decreases since the bubble grows faster than the center of the bubble moves upwards. After three to four seconds the shape of the bubble

becomes drop like due to its upward movement. At  $t = 4.24$  sec. a second nucleus becomes visible at the position where the first nucleus was formed. Bubble 2 is probably formed from residual vapor left over in the wake of bubble 1. This sequence is repeated another time before bubble formation above the tube stops in this part of the tube for a longer period.

Effects like this were observed regularly. After some period of time a first nucleus is formed spontaneously in the superheated liquid layer without contact to the heated surface. Vapor residuals in the wake of the resulting bubble form a nucleus for subsequent bubbles – something that might be considered an activated nucleation site in a superheated liquid layer. After a number of bubbles



**Figure 7:** Formation of bubbles at the side of the test tube. Both bubbles are formed from a nucleation side on the surface of the test tube.

has been formed, the temperature of the liquid layer has dropped below local saturation temperature, whereby the local saturation temperature has increased due to decreasing n-pentane concentration at the same time – the process stops. A more detailed analysis of the measured temperatures supports this thesis. The temperatures shown in Figure 5 are averaged over time. Actually temperatures measured close to the surface oscillate by up to  $\pm 5$  K over long time intervals ( $> 100$  sec.). However, recorded temperatures were not yet synchronized with observed periods of bubble formation.

Once stable conditions are found in the described experiment, none of the bubbles formed above the test tube ever had contact to the heated surface. All of them were formed in the superheated liquid layer. Obviously, the surface is covered by a layer of almost pure silicon oil, in which no further bubble formation is possible. Heat from the test tube is transported through this liquid layer to regions with higher concentration of n-pentane, where bubbles can be formed. This experimental finding supports the thesis by Gerber and Span (2005) that a second form of critical behavior leading to high superheats at the wall can be observed for boiling highly viscous mixtures. However, experiments also indicate that defining a critical heat flux for this effect will become difficult.

Figure 7 shows bubbles formed at the side of the test tube almost at the same time as those shown in Figure 6, in a position shifted about 15 mm in axial direction. At  $t = 6.12$  sec. bubble 1 is formed from a nucleation site on the wall of the test tube. Within less than one second the bubble grows to a diameter of about 7.5 mm. After about three seconds it starts to detach. 7 seconds after bubble 1 a second bubble is formed from the same nucleation site.

Obviously, not the whole test tube is shielded by an accumulation of non-volatile component. While bubbles are formed in a superheated liquid layer above the tube, nucleation still takes place on the surface at the side of the tube and also underneath of it. Mixture with average pool composition is transported to the tube from underneath by convection. Above the tube, the non-volatile component accumulates and forms an isolating layer. Yet no data are available on the peripheral surface-temperature distribution, but Figure 5 shows temperature gradients close to the surface, which are larger beside and under the test tube, indicating better heat transfer in these regions.

Figures 6 and 7 also show the effect of condensation of bubbles getting into subcooled liquid regions. Bubbles formed above the tube maintain constant size until their top reaches a point about 10 mm above the heated surface ( $t = 3.88$  sec.). Beyond that point condensation can be observed – the bubble starts to shrink. However, this effect should have no impact on the liquid layer formed above the tube. Bubbles formed at the side of the tube get in touch with subcooled liquid layers much earlier. They start to shrink the moment they start to detach ( $t = 9.60$  sec.). Thus, bubble formation and heat transfer beside and under the test tube might be substantially different in a properly equilibrated pool. The question whether and if yes at which “critical” heat flux the observed layer of non-volatile component can cover the tube completely cannot be answered before the apparatus modified for highly viscous mixtures becomes available.

## 5. Summary

A concept to analyze heat and mass transfer in highly viscous mixtures has been described. The objective of this investigation, in addition to quantifying heat transfer, is the description of effects that are unique for boiling highly viscous mixtures.

As a first step into the project, a standard apparatus for measuring heat transfer in an equilibrated pool is modified for measurements on extremely wide-boiling mixtures. The design of the modified apparatus has been described. Additional modifications necessary to adapt a standard apparatus to wide-boiling highly-viscous mixture were briefly discussed.

First results were presented for mixtures of n-pentane and silicon oil with a viscosity of  $10^6$  mPa s boiling in an open subcooled pool at ambient pressure. A new mechanism was observed for bubble formation above the test tube. While bubbles at the side of the test tube are still formed at nucleation sites on the heated surface, bubbles are formed in a superheated liquid layer without contact to the wall above the tube. The upper part of the tube is covered by a layer of almost pure silicon oil, in which bubble formation cannot take place.

However, bubble formation below and at the side of the test tube may be affected by the fact that the pool is subcooled. The same is true for the measured heat transfer coefficient. The modified standard apparatus under construction is required to report quantitatively reliable results on heat transfer coefficients and on critical heat fluxes separating boiling with nucleation on the surface of the test tube from states with nucleation in a superheated liquid layer.

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