

# Influence of critical phenomena on the heat transfer to binary mixtures

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**Abstract**—Free convective heat transfer to mixtures of ethane and n-heptane are studied experimentally. A horizontal platinum wire is used in the investigations. During the measurements the pressure is kept above the critical pressure, and the bulk temperature below the critical temperature of the mixture. For pressures below the maximum pressure of the phase envelope, i.e. in the region where retrograde condensation could occur, a strong increase of the heat transfer coefficient is observed coinciding with the onset of retrograde condensation. Visual observations show highly turbulent boiling-like behaviour of the flow pattern at the wire during this mode of heat transfer. For high-temperature differences the heat transfer mechanism is found to be similar to film boiling for pressures not too close to the critical point. If the pressure is close to the critical point the heat transfer mechanism for high-temperature differences changes and resembles turbulent free convective heat transfer.

## INTRODUCTION

FREE CONVECTIVE heat transfer to pure fluids close to the critical point has been investigated in numerous studies [1-7]. Reviews are given by Hendricks *et al.* [8] and Hall [9]. Theoretical investigations [10] for laminar free convection confirmed the influence of fluid properties on the heat transfer enhancement at the critical point. In recent investigations [11-14] forced and free convective heat transfer to binary mixtures were studied. Jump and Marshall [13] used azeotropic refrigerant mixtures in their studies. Similar to pure fluids an increase of the heat transfer coefficient for small temperature differences was found. Jones *et al.* [14] performed experiments with mixtures of carbon dioxide and n-decane. In all experiments an enhancement of the heat transfer was observed.

In the present investigation free convective heat transfer to mixtures of ethane-n-heptane in the critical region was studied. A thin wire was used in the measurements. The goal of the experiments was to investigate the enhancement of heat transfer at the critical point and the influence of retrograde condensation.

## FUNDAMENTALS

Similar to pure fluids the fluid properties of binary mixtures exhibit strong changes or peaks close to the critical point of the mixture. For each given composition of a binary fluid a critical point exists. Therefore, as shown in Fig. 1, the critical points of a binary mixture represent the critical locus line if plotted in a  $p$ - $T$  diagram. For most mixtures the critical locus line runs continuously between the critical points of the pure components which are the end points of the vapour pressure curves of the pure components [5]. It

should be noted that critical points of the mixture can be at pressures or temperatures which are higher than the critical pressure or temperature of the pure components. Additionally, Fig. 1 shows the phase envelope of a mixture with given composition  $x_m$ . The two-phase region is bounded by the dew-point line and the boiling-point line which meet at the critical point  $c_m$  of the mixture. For pure fluids no vapour-liquid phase transitions are possible for pressures above the critical pressure and for temperatures above the critical temperature. As can easily be seen from Fig. 1, for a mixture with given composition  $x_m$  two phases can coexist for pressures up to  $p_{max}$  and for temperatures up to  $T_{max}$  which are above the critical pressure and temperature of the mixture. Therefore, in contrast to pure fluids in mixtures two phases can coexist above the critical point.

These properties of mixtures cause so-called retrograde phenomena which were first described in detail

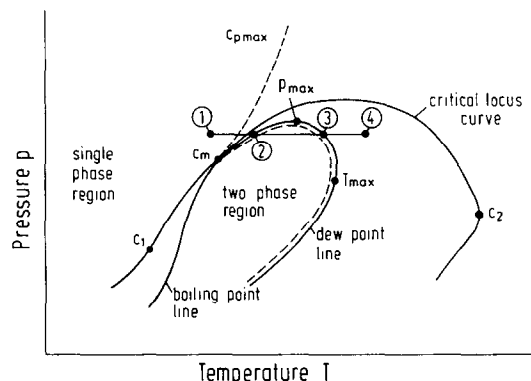


FIG. 1. Schematic phase diagram of a binary mixture in the  $p$ - $T$  plane.

## NOMENCLATURE

$c_p$	isobaric specific heat capacity [kJ kg <sup>-1</sup> K <sup>-1</sup> ]
$n$	exponent defined by equation (2) [—]
$p$	pressure [bar]
$\dot{q}$	heat flux [W m <sup>-2</sup> ]
$\Delta T$	temperature difference [K].

## Greek symbol

$\alpha$	heat transfer coefficient defined by equation (1) [W m <sup>-2</sup> K <sup>-1</sup> ].
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## Subscripts

b	bulk
c	critical
m	mixture
max	maximum value
w	wire.

by Kuenen [16]. Retrograde condensation occurs if, as shown in Fig. 1, the temperature of a mixture is increased at constant pressure from point 1 to point 4. At point 1 a single phase exists. Increasing the temperature of the mixture the dew-point line is reached. When at point 2 the dew-point line is traversed from the single phase to the two-phase region, a liquid phase occurs. Increasing the temperature further, the dew-point line is traversed again at point 3, now from the two-phase to the single phase region. Therefore, the liquid phase disappears and again only a single phase exists. While continuously increasing the temperature first a liquid phase occurs which later disappears again on further increasing temperature. The phenomenon of retrograde condensation can also occur in heat transfer processes. If the bulk condition of a binary mixture is such that the temperature and pressure of the mixture are represented by point 1 in Fig. 1 and the condition at a heated surface by point 4, retrograde condensation occurs in the thermal boundary layer. In this case a twofold phase change occurs in the thermal boundary layer. This should introduce disturbances and therefore enhance the heat transfer.

In Fig. 1 the locus of the maximum value of the specific heat capacity  $c_{p\max}$  in the  $p$ - $T$  plane is also plotted schematically as a broken line. The curve which is also called the pseudocritical curve originates at the critical point of the mixture and exhibits a positive slope and curvature [17]. This behaviour is similar to pure fluids where the pseudocritical curve is a smooth continuation of the vapour-pressure curve. The maximum values of the coefficient of thermal expansion have an analogous behaviour. Therefore, density decreases rapidly for temperatures above the pseudocritical temperature.

An estimate of the critical data of the ethane-n-heptane mixtures used in the investigation yielded  $T_c = 46$  C,  $p_c = 57$  bar and  $p_{\max} = 63$  bar for a mole fraction of 97.3% ethane and  $T_c = 45$  C,  $p_c = 55.5$  bar and  $p_{\max} = 61$  bar for a mole fraction of 97.6% ethane. Although vapour-liquid equilibrium data for the binary mixture ethane-n-heptane exist [18, 19] a

quantitative description of all mixture properties in the critical region, needed for heat transfer calculations, is difficult. Using correlations or equations of state for calculating fluid properties yield unsatisfactory results mainly due to insufficient experimental property data. Nevertheless, the qualitative description of the fluid properties of ethane-n-heptane as given in Fig. 1 is satisfactory to describe the observed heat transfer phenomena.

## EXPERIMENTAL APPARATUS AND PROCEDURES

The experiments were carried out with an apparatus shown schematically in Fig. 2. The main components of the apparatus are a pressure cell with a thin wire mounted horizontally inside the cell.

The pressure cell has a volume of about 1.3 l and is built of stainless steel. It consists of two cylinders, a horizontal cylinder upon which a second cylinder is welded vertically. The vertical cylinder houses a piston which can be moved from the outside with an elevating screw. In the flanges of the horizontal cylinder two Pyrex windows are installed to allow for visual observation of the wire.

In the horizontal part of the pressure cell an electrically heated platinum wire of 0.1 mm diameter is mounted horizontally serving as a heating surface. As an electrical source a high current d.c. power supply is used. The pressure in the cell is measured with a high precision Bourdon manometer and with a pressure transducer. The bulk temperature in the cell is determined with a platinum resistance thermometer. The fluid in the cell can be stirred with a magnetic stirrer.

The cell is insulated and can be thermostated with air. Capillary tubes connect the cell with valves used for evacuating and filling the cell with the test mixture. After evacuating the cell, each of the components was filled in separately. To facilitate the filling the cell can be cooled below the ambient temperature with a cryostat. The amount of substance filled in was determined by weighing the sample vessels before and

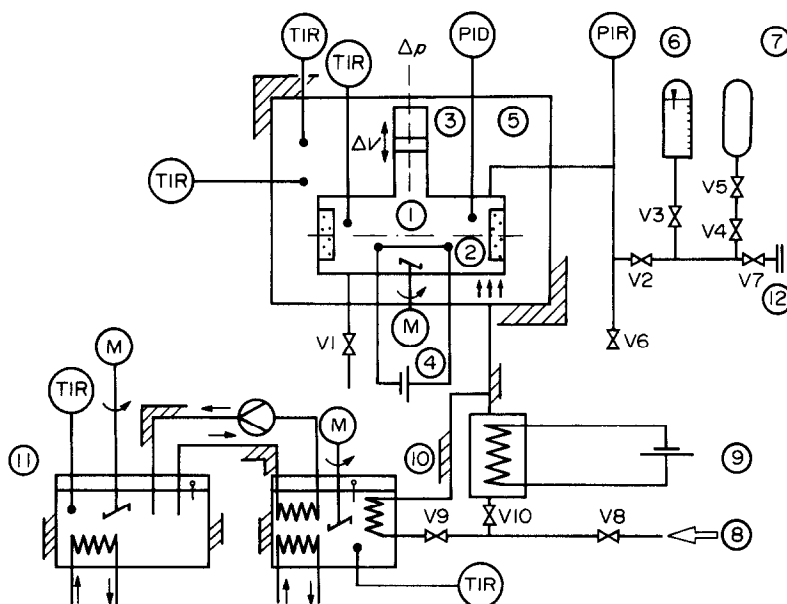


FIG. 2. Experimental apparatus: 1, pressure cell; 2, platinum wire; 3, piston to change volume of cell; 4, stirrer; 5, with air thermostated enclosure; 6, sample vessel; 7, sample vessel; 8, air supply; 9, air heater; 10, air heat exchanger; 11, cryostat; 12, vacuum.

after filling. The composition could then be determined with an estimated accuracy of 0.05%.

After filling the test cell with the mixture, the temperature of the cell was adjusted to a value below the critical temperature of the mixture. When steady state was reached the pressure in the cell could be set to a value above the critical pressure of the mixture. This was accomplished by changing the volume of the test cell by moving the piston in the cell with the elevating screw.

Measurements were taken at constant pressure. The electrical power input was increased in steps up to  $150 \text{ kW m}^{-2}$ . Because of the low wire surface the overall heat input was small in relation to the large volume of the test mixture so that the change of temperature and pressure in the cell could be neglected.

The reported heat transfer coefficients are calculated from

$$\alpha = \frac{\dot{q}}{T_w - T_b} \quad (1)$$

Assuming no heat losses, the electrical power input equals the heat transferred from the wire to the mixture. The electrical power input was determined from the voltage drop across the wire and across a precision resistor. The bulk temperature  $T_b$  is measured with a platinum resistance thermometer. The wire temperature  $T_w$  was calculated from the resistance of the wire at bulk temperature and the known coefficient of resistivity for platinum. Errors in the heat transfer coefficient were estimated to be less than 4% for temperature differences of 20 K. The errors

increase for smaller temperature differences and reach 12% for temperature differences of 1 K.

## EXPERIMENTAL RESULTS AND DISCUSSION

### Heat transfer with retrograde condensation

Experimental results for a mixture of 97.6% ethane and 2.4% n-heptane are shown in Fig. 3. The bulk temperature was  $38.5^\circ\text{C}$ , the pressure in the cell 56.6 bar. The data points were taken at constant current through the wire which was increased in steps. For each heat flux from the wire to the mixture one data point was taken. In Fig. 3 the heat flux is plotted as a function of the temperature difference between the wire and bulk temperature. Three different characteristics of the dependency of the heat flux on the temperature difference can be distinguished. For small heat flux values the temperature difference increases proportional to the heat flux. From a heat flux of  $25 \text{ kW m}^{-2}$  on, the temperature difference remains constant at 9 K up to a heat flux of  $50 \text{ kW m}^{-2}$ . For high heat flux values the temperature difference increases again.

The different functional dependencies of the heat flux on the temperature difference can be explained when the corresponding phase behaviour of the mixture is taken into consideration. Together with the visual observations made during the measurements, the phenomena can be made clear. In Fig. 1 the state transition from point 1 to point 4 shows schematically the state of the fluid at the wire surface during the measurements. When the wire is not heated, pressure

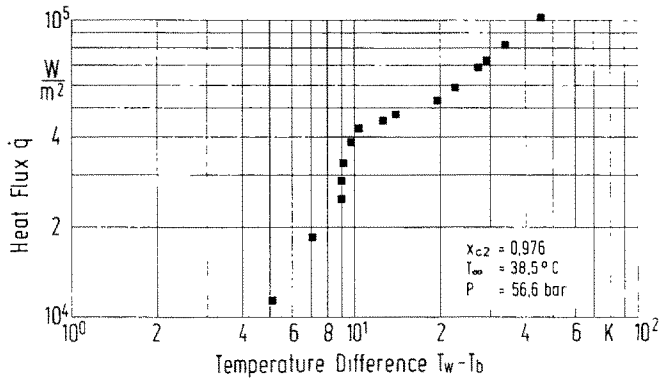


FIG. 3. Heat flux vs temperature difference.

and temperature are resembled by point 1. When a current is applied to the wire, the condition at the wire surface moves along an isobar from point 1 to point 4. As soon as the wire temperature is at point 2 retrograde condensation can occur. Between point 2 and point 3 retrograde condensation occurs which enhances the heat transfer drastically. Therefore, as shown in Fig. 3 the temperature difference remains at 9 K even when the heat flux is increased. The heat transfer is enhanced for the data points in Fig. 3 where the wire temperature corresponds with the temperatures between points 2 and 3 in Fig. 1.

The enhancement of heat transfer for temperature differences between 9 and 20 K due to retrograde condensation was confirmed by visual observations of the flow pattern. The enhancement coincided with a sudden change of the flow pattern as shown in Fig. 9. The left part of the photograph shows a highly agitated flow pattern above the dark horizontal line representing the wire. The agitated flow pattern was observed when retrograde condensation occurred. The right part of the photograph shows a region above the wire with no turbulence. It should be mentioned that the photograph does not show a stable condition. The transition zone between laminar and turbulent flow moved from left to right along the wire when the photograph was taken. The agitated flow pattern started at a single point and then proliferated along

the wire in analogy to boiling which can also start at a single nucleation site and subsequently spread over the heated surface. Boiling-like phenomena were reported earlier by Nishikawa *et al.* [6] and by Knapp and Sabersky [5] for pure substances above their critical point. The photographs for boiling-like flow in the work of Nishikawa *et al.* [6] and of Knapp and Sabersky [5] show a close resemblance to Fig. 4.

The enhancement of heat transfer in the temperature range where retrograde condensation occurs, can be seen in Fig. 4 where the heat transfer coefficient is plotted as a function of the wire temperature. The same dependencies can be observed as in Fig. 3. For the first data points the heat transfer coefficient increases with wire temperature, then for a temperature range corresponding to the temperatures of points 2 and 3 in Fig. 1 the heat transfer coefficient reaches peak values above  $4000 \text{ W m}^{-2} \text{ K}^{-1}$ . For high wire temperatures the heat transfer coefficient decreases slowly with the wire temperatures.

#### Heat transfer in the critical region without retrograde condensation

In Fig. 5 results are plotted for different system pressures. The ethane-n-heptane mixture contained 97.6% ethane. The bulk temperature was  $38.5^\circ\text{C}$ . The pressure levels cover a range from pressures where retrograde condensation can occur to pressures where

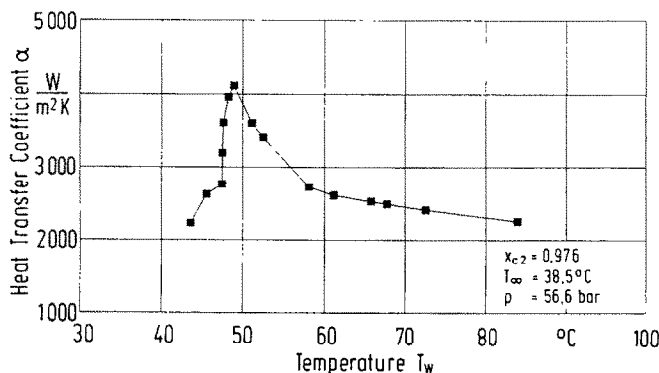


FIG. 4. Flow pattern at wire.

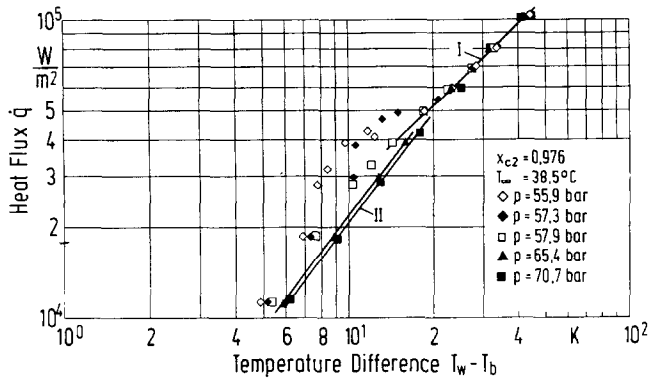


FIG. 5. Heat transfer coefficient vs wire temperature.

no phase change is possible. For the data points at pressures of 55.9 and 57.3 bar retrograde condensation occurs. The curves for these two pressures are similar to the curve in Fig. 3. For pressures of 57.9, 65.4 and 70.7 bar no retrograde condensation occurs. As shown in Fig. 6, the data points can be approximated by straight lines. For temperature differences up to 20 K the heat flux depends on the pressure, whereas for temperature differences above 20 K the data points can be approximated by a single line. For pressures where retrograde condensation occurs, the data points for temperature differences below and above the retrograde region show a similar behaviour to the data points with no retrograde condensation.

The slope of the lines in Fig. 5 change at temperature differences between 10 and 20 K which is attributed to the fact that the pseudocritical temperature is reached where fluid properties change strongly. In analogy to the  $c_{pmax}$  curve in Fig. 1 the change of slope occurs for higher pressures at higher temperatures. In free convection and pool boiling heat transfer the dependency of the heat flux on the temperature difference can be described by power laws of the type

$$\dot{q} \sim \Delta T^n \quad (2)$$

where the exponent  $n$  depends on the mode of heat

transfer. Therefore, calculating the exponent  $n$  from the lines shown in Fig. 5 the mode of heat transfer can be deduced. A least squares fit of the data points (line I) with temperature differences above 20 K yields  $n = 0.86$ . This value is close to  $n = 4/5$  which is found in the literature for film boiling [20]. The sudden decrease of density for wire temperatures above the pseudocritical temperatures could explain the formation of a low density layer around the wire analogous to a layer of vapour formed in film boiling. This explanation was confirmed by the visual observation which showed turbulence only at a distance above the wire and no turbulence at the wire. The data points on line II are below the pseudocritical temperature. Therefore, values of the specific heat capacity and density are much higher than for the data points on curve I. A least squares fit for line II yields  $n = 1.3$  which is close to the value of  $n = 4/3$  for turbulent free convection.

For the same data points as in Fig. 5 the heat transfer coefficient is plotted as a function of the wire temperature in Fig. 6. For the pressures where retrograde condensation occurs a strong increase of the heat transfer coefficient can be observed. For pressures of 57.9, 65.4 and 70.7 bar no phase change occurs. The heat transfer coefficient reaches a maximum and decreases slowly for higher temperatures of the wire. The temperature for which the

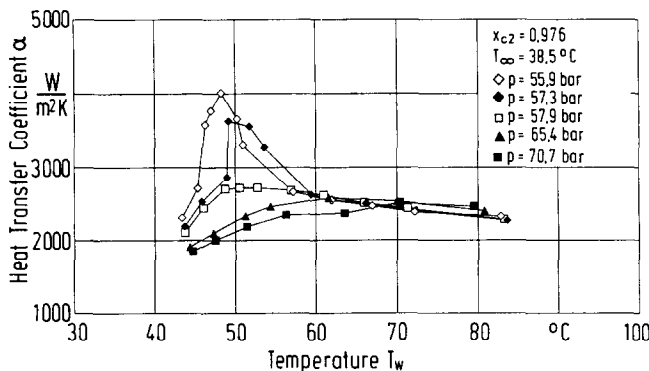


FIG. 6. Heat flux vs temperature difference.

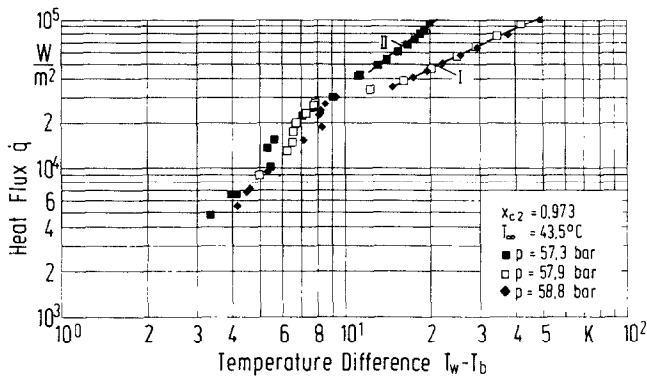


FIG. 7. Heat transfer coefficient vs wire temperature.

maximum heat transfer coefficient occurs is a function of the system pressure analogous to the behaviour of the pseudocritical temperature.

#### Heat transfer close to the critical pressure

Figure 7 shows measurements for three different pressure levels for a mixture of 97.3% ethane and 2.7% n-heptane. The bulk temperature was 43.5°C for all three data series. The heat flux density is plotted as a function of the temperature difference. As before different types of dependencies of the heat flux on the temperature difference can be distinguished. The data series at a pressure of 57.9 and 58.8 bar are similar to those in Fig. 5. The curve for a pressure of 57.3 bar is above these data points for temperature differences above 10 K. For temperature differences up to 9 K the curves show the same behaviour.

Approximating the data points for high-temperature differences with the straight lines I and II the mode of heat transfer can be concluded from their slope. The slope of curve I yields values for  $n$  of the order of 4/5 as in Fig. 5. The slope of curve II is of the order of 1.35 which is close to the value of 4/3 observed in turbulent free convection, i.e. the turbulent flow pattern observed when the wire temperature is in the retrograde region is extended to higher wire temperatures. This turbulent free convection occurred for pressures close to the critical

point. The visual observation showed a turbulent flow pattern at the wire. For a pressure of 57.3 bar the heat transfer coefficient increases with the temperature difference as can be seen in Fig. 8 where the heat transfer coefficient is plotted as a function of the wire temperature. At pressures of 57.9 and 58.8 bar the data series are analogous to the data shown in Fig. 7. When the wire temperature has a value in the phase envelope a strong increase of the heat transfer coefficient is observed. For a pressure of 57.3 bar the heat transfer coefficient increases to over 4500  $\text{W m}^{-2} \text{K}^{-1}$ .

#### Visual observations

Several authors [12, 14] reported liquid flowing down from the wire. In the investigation reported here for mixtures of ethane-n-heptane no downward flow was observed. Although, downward puffs from the wire were seen occasionally as reported by Hernandez and Marshall [12]. In some of the measurements droplets forming at the bottom of the wire and disappearing again could be seen. It can be assumed that the occurrence of liquid flowing downward depends on the amount of liquid formed during retrograde condensation. This in turn would depend on the phase behaviour of the binary mixture used. Therefore, more accurate fluid property data would allow the study of this phenomena.

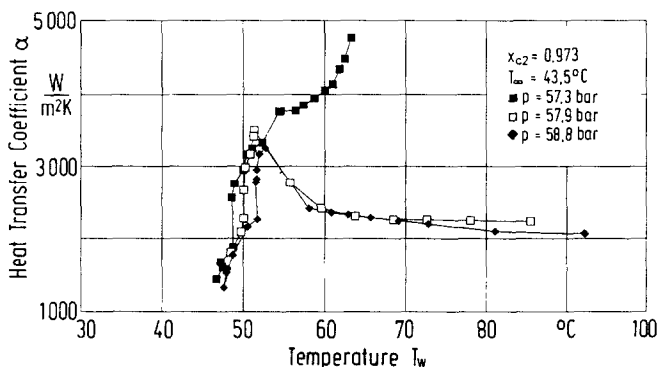


FIG. 8. Heat flux vs temperature difference.

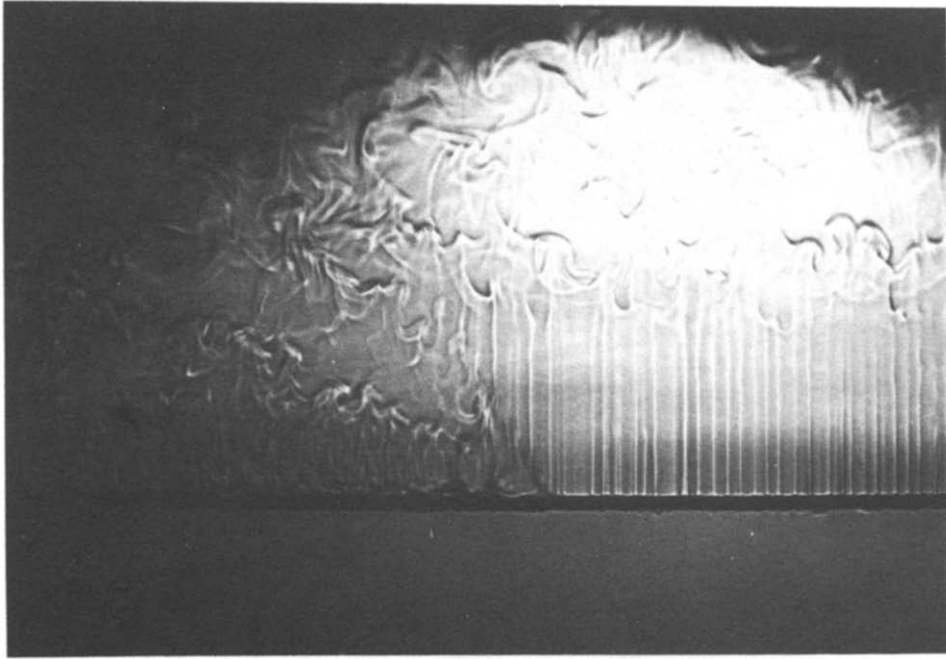


FIG. 9. Heat transfer coefficient vs wire temperature.

### SUMMARY AND CONCLUSIONS

Free convective heat transfer to binary mixtures above their critical pressure was studied experimentally. A more than twofold enhancement of the heat transfer coefficient to values over  $4000 \text{ W m}^{-2} \text{ K}^{-1}$  at temperature differences of  $5 \text{ K}$  was found due to the occurrence of retrograde condensation. Visual observations showing boiling-like phenomena confirmed the influence of retrograde condensation. When the wire temperature reaches the retrograde region, a sudden change of the flow pattern from laminar to turbulent boiling-like flow is observed.

For high-temperature differences and pressures above  $p_{\text{max}}$  two heat transfer modes were found. If the wire temperature is below the pseudocritical temperature, the dependency of the heat flux on the temperature difference resembles turbulent free convection. For wire temperatures above the pseudocritical temperature the heat transfer mechanism is similar to film boiling. The strong decrease of density at the pseudocritical temperature causes the formation of a low density layer around the wire analogous to a layer of vapour formed in film boiling. The visual observations showed turbulence starting at a distance above the wire with no turbulence at the wire.

Close to the critical point the heat transfer phenomena were found to be similar to turbulent free convection. In this case the turbulent flow pattern observed in the retrograde region was extended to higher temperature differences, i.e. the turbulence induced by retrograde condensation persisted for wire temperatures above the retrograde region. The heat transfer coefficient increased with the temperature difference and reached values of over  $4500 \text{ W m}^{-2} \text{ K}^{-1}$ . In

order to describe the observed phenomena quantitatively, a better understanding of the fluid properties of binary mixtures in the critical region would be necessary.

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### INFLUENCE DES PHENOMENES CRITIQUES SUR LE TRANSFERT THERMIQUE DANS LES MELANGES BINAIRES

**Résumé**—On étudie expérimentalement la convection thermique naturelle dans des mélanges d'éthane et de n-heptane. Un fil horizontal de platine est utilisé. Pendant les mesures la pression reste au dessus de la pression critique et la température moyenne au dessous de la température critique du mélange. Pour des pressions inférieures à la pression maximale de l'enveloppe de phase, par exemple dans la région où la condensation rétrograde peut apparaître, un fort accroissement du coefficient de transfert est observé en même temps que la condensation rétrograde. Des observations optiques montrent une ébullition fortement turbulente autour du fil pendant ce mode de transfert. Pour des différences de température élevées le mécanisme de transfert de chaleur est semblable à l'ébullition en film pour des pressions pas trop proches du point critique. Si la pression est voisine du point critique, le mécanisme de transfert thermique pour les grandes différences de température change et ressemble au transfert thermique turbulent de convection naturelle.

### EINFLUSS KRITISCHER PHÄNOMENE AUF DEN WÄRMEÜBERGANG BINÄRER GEMISCHE

**Zusammenfassung**—Der konvektive Wärmeübergang an binäre Ethan-n-Heptan Gemische wurde experimentell untersucht. In den Versuchen wurde ein horizontaler Platindraht als Heizfläche verwendet. Während der Messungen war der Druck größer als des kritische Druck des Gemisches, die Temperatur des ungestörten Fluids war unterhalb der kritischen Temperatur des Gemisches. Für Drücke unterhalb des maximalen Druckes der Phasengrenzlinie, d.h. innerhalb des retrograden Gebietes, wurde gleichzeitig mit dem Auftreten von retrograder Kondensation eine starke Erhöhung des Wärmeübergangs beobachtet. Visuelle Beobachtungen zeigten eine hoch turbulente siedeähnliche Strömungsform am Draht während dieses Vorgangs. Bei sehr hohen Temperaturdifferenzen zwischen Draht und Fluid änderte sich der Wärmeübergangsmechanismus und ähnelte Filmsieden, solange der Druck nicht allzu nahe am kritischen Druck lag. Für Drücke nahe am kritischen Druck ergab sich ein Wärmeübergangsverhalten wie bei turbulenter freier Konvektion.

### ВЛИЯНИЕ КРИТИЧЕСКИХ ЯВЛЕНИЙ НА ПЕРЕНОС ТЕПЛА В БИНАРНЫХ СМЕСЯХ

**Аннотация**—Экспериментально исследуется конвективный перенос тепла в смесях этана и н-гептана. В исследованиях используется горизонтальная платиновая проволока. В ходе измерений давление сохраняется постоянным, выше критического, а среднemasсовая температура—ниже критической температуры смеси. При давлениях ниже максимального давления, соответствующему равновесию двух фаз данной смеси, т.е. в области, где может происходить ретроградная конденсация, наблюдается сильное увеличение коэффициента теплообмена, совпадающее с возникновением ретроградной конденсации. При визуальных наблюдениях обнаружено высокотурбулентное кипение, аналогичное структуре течения у проволоки при данном режиме теплопереноса. В случае больших разностей температур механизм теплопереноса оказывается сходным с пленочным кипением при давлениях, вдали от критической точки. Если же давление приближается к критической точке, механизм теплопереноса при больших разностях температур изменяется, и он становится похожим на турбулентный свободноконвективный.