Wetting Effects on Boiling Heat Transfer: the Copper-Stearic Acid System

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In order to study effects of wetting on heat transfer in the nucleate boiling regime, stearic acid was boiled in contact with different crystal planes of single crystals of copper. One crystal plane being wetted by the acid more completely than the other, they are called *wetted* and *nonwetted* surfaces. In the region of low heat flux, where heat transfer is primarily nonboiling natural convection, the nonwetted crystal required higher values of temperature difference than the wetted crystal for the same flux. At high values of heat flux, though not in the vicinity of the critical temperature difference, the situation was reversed; that is, the nonwetted surface required lower temperature difference than the wetted surface.

In the present studies, stearic acid was boiled at about 465°F., corresponding to a pressure of 17 mm. of mercury. Heat flux ranged from 3,450 to 63,300-B.t.u./(hr.)(sq. ft.), and temperature difference between the copper crystal and the stearic acid ranged from 38° to 132°F. The corresponding range of heat transfer coefficient was from 91 to 510 B.t.u./(hr.)(sq. ft.)(°F.).

An influence of wetting on heat transfer has long been recognized in the boiling processes (*1, 2, 3*), and recently an influence of wetting has been suspected in convective studies with certain liquid metals such as mercury (*4* to *7*). The idea is that, if a surface is not wetted by a liquid, the thermal resistance across the interface would be greater than if the surface is wetted by the liquid. An additional consideration in the boiling mechanism is the influence that this condition has on the ease with which a vapor bubble may be formed and removed from the interface.

McAdams (*3*) and Jakob (*2*) have pointed out some of the qualitative effects of wetting on boiling heat transfer. In boiling studies, some observers have used detergents to change the surface tension of the liquid (*8*). In nonboiling convective heat transfer, small additions of sodium have been made to mercury in order to promote wetting (*4, 6*), and different surface materials have been used with mercury, such as stainless steel, which is not wetted, and copper, which is wetted (*7*). Nonboiling convective heat transfer to liquid sodium has also shown some evidence of wetting effects at a heat transfer surface (*9*). Thermal resistances across a sodium-stainless steel interface have been measured (*10, 11*), and measurements of electrical resistance across liquid-solid interfaces have also been made with liquid mercury and liquid sodium (*12, 13*) in an effort to learn more about the wetting mechanism. In these cases reported in the literature, *wetting* and *nonwetting* are achieved by one of the following methods:

1. Addition of agents to the liquid to alter its surface tension or increase wetting.
2. Coating of the solid surface with a film to change the nature of the interface.
3. Alternate use of differing solid materials with the same liquid.
4. Alternate use of differing liquids with the same solid surface.

Another method has been employed in the present work, based on observations of Gwathmey, Leidheiser, and Smith (*14*), who, examining the chemical action of oils on single copper crystals, found that, under certain conditions, the degree of wetting of a solid by a liquid depended on the orientation of the crystal lattice at the liquid-solid interface. In particular, they found that at 200°C. only the 110* faces of a crystal were wetted by stearic acid in air, and that the 100* faces were not. These observations suggest the mode of approach for the present experiments: to use the same material (copper) in contact with the same liquid (stearic acid) but to achieve different degrees of wetting by having different crystal faces in contact with the liquid at the interface.

MECHANISM OF BOILING HEAT TRANSFER

Various general descriptions of the boiling process may be found in the work of such writers as Bosworth (*1*), and McAdams (*3*), and Jakob (*2*). Hawkins (*15*) prepared an extensive review of the literature on boiling heat transfer up to 1950. Since that time many papers on boiling have appeared, some of which seem to be pertinent to the present problem.

The process involving formation of discrete bubbles at the interface is referred to as *nucleate* boiling. The term implies the existence of *vaporization nuclei* which promote formation of individual bubbles. The exact nature of the nuclei is somewhat in doubt, but it may be that they are gas molecules adsorbed at the solid surface, gas molecules absorbed in the liquid, gas molecules generated by chemical reaction between the solid and liquid, or small solid particles suspended in the liquid. However, the nuclei may be nothing more than cavities or roughness elements in the surface which favor the formation of bubbles or differing force fields arising from differing atomic lattice orientations at the surface.

Corty and Foust (*16*), investigating the influence of *degree of wetting* and *surface roughness* on nucleate boiling, concluded that the size and shape distribution of roughness elements on the surface and the contact angle of the bubble influenced the temperature difference required to achieve a particular heat flux, as well as the number of *active* centers (locations on the surface which favor repeated bubble formations). It might be pointed out here that there is a connection between contact-angle measurements and surface roughness. Wenzel (*17*) has proposed that the important aspect of roughness as it pertains to wetting is the ratio of actual surface area to projected area, rather than the mean height of roughness elements which is determined from surface profiles. He refers to findings of Bikerman which indicated the same contact angle for stainless steel plates having heights of roughness elements differing by a factor of over a hundred. The possibility also exists that actual force fields are on a microscopic scale but that contact angles must be on a much larger scale and hence may not faithfully reflect the important atomic or molecular interaction.

It may well be that the true effect of surface roughness is related to the atomic population density of the surface material. In a polycrystalline surface with random crystal orientations, if force field can be related to the number of atoms/unit of surface area, it is apparent that different crystal faces may have different attractive forces for the same liquid. Thus, the *active* center may be merely the location at which crystal-lattice orientation provides the least attraction for the liquid. Roughness may serve the function of providing only more of such crystal faces for a unit of projected area (though perhaps no more for a unit of actual area). Later in the report is a description of an effort to learn more about this effect of crystal orientation.

Jakob (*2*) has reported data on the frequency of formation of discrete bubbles at particular spots on a solid surface. Apparently the formation process is cyclic and the frequency is related to the ultimate size attained by the bubble just before it leaves the surface. The *leaving* size of the bubble is believed to be related to the interfacial attraction between the liquid and solid, the surface tension of the liquid, the buoyant force arising from the

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*Miller indexes.

difference in density of the liquid and vapor, and perhaps other factors.

Some of the most penetrating comments on the boiling mechanism have been presented by Larson (*18*, *19*), summarizing his earlier investigations (*20*) and expanding his concept of an *ebullator* theory. In developing this theory, Larson has taken into account attractive or Van der Waals' forces, repulsive forces related to overlap of atomic or molecular electron orbits, and relative atomic spacing of the solid and liquid. He suggested further that the influence of wettability would show in a family of curves, with the maximum flux and the corresponding temperature difference both increasing as wettability increases. It may be that experimental data on maximum heat flux obtained in nucleate boiling in geometrically similar systems can be used to estimate the degree of wettability.

THE WETTING OF COPPER BY STEARIC ACID

Before proceeding further in the discussion of wetting effects, one should define what is meant by the term *wetting* and the associated concept of *contact angle*. *Wetting* is considered to represent closeness of contact between a solid and a liquid. Three typical shapes of water-vapor bubbles on a heated surface are indicated by Jakob (*2*) to describe nonwetting, partial wetting, and wetting of the solid by the liquid. Spreading of a liquid drop on a solid is also indicative of wetting. If the drop spreads out over the surface, the surface is said to be wet. If the drop remains as a spheroid on the surface, the surface is not wet. At the solid-liquid-vapor interface the degree of wetting is ordinarily related to the angle (measured through the liquid) which the liquid-vapor interface makes with the solid-liquid interface at the intersection with the solid-vapor interface. A contact angle of zero implies complete wetting of the solid by the liquid, 180 deg. implies complete nonwetting, and any intermediate angle would describe degree of partial wetting. Gregg (*21*) and a number of other writers have extensively discussed wetting and contact angles.

As mentioned previously, the objective of the present work is to study wetting effects on boiling. The intent is to perform boiling heat transfer studies wherein changes in the degree of wetting are affected between the same solid and liquid. In the study of Gwathmey, Leidheiser, and Smith (*14*) on the chemical action of oils on single metal crystals, it was found that stearic acid would preferentially wet 110 planes on a spherical single crystal of copper, provided that the crystal had been alternately immersed in stearic acid and exposed to air for a certain period of time. In an atmosphere of hydrogen, this effect was not exhibited, an indication that the presence of oxygen as an oxide layer or complex was essential in the mechanism. They also observed differences in rates of oxidation at different crystal planes as evidenced by the rate of change in color patterns on surfaces. The 100 planes apparently were the most active with respect to oxidation. Immersed in stearic acid, the crystal showed etch patterns which caused striated roughness at the 110 planes and smooth regions at the 100 and 111 planes. With respect to roughness as a factor in the wetting studies, they concluded that it had an influence but not a controlling one. One of their reports includes a detailed discussion of their results with respect to the present theory of the influence of crystal plane on surface reactions (*22*) and should be consulted for a more complete understanding of the status of the problem. Briefly, it appears that arrangement of the atoms at the surface, including the number/unit area (population density), plays an important part in controlling reaction rates. Theoretical studies did not enable predictions for rates of oxidation, and simple energy concepts based on spacing of the atoms appeared to be of little value for such predictions.

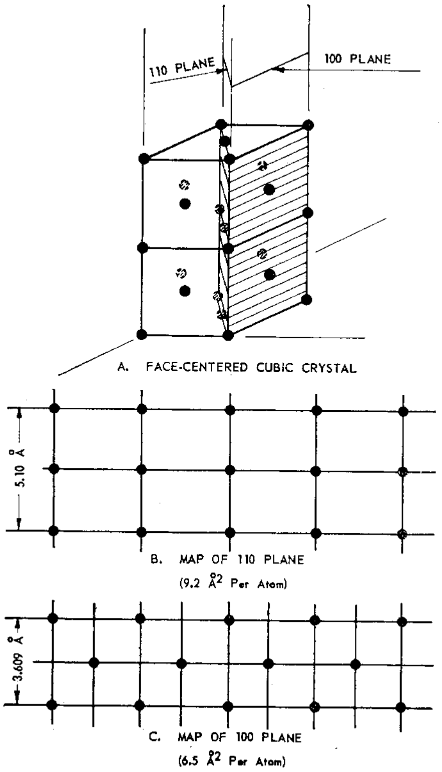

Fig. 1. Details of copper single crystal.

The coexistence of differing roughness and differing wetting at differing crystal planes confuses the picture by preventing a separation of the variables. With respect to the influence of roughness on wetting, there are some interesting studies by Ray and Bartell (*23*) which show that contact angles are influenced by microroughness of extremely fine structure, perhaps even molecular in size. One might conclude that, from the difference in roughness alone, one should expect a difference in wetting of copper by stearic acid at different crystal faces.

Figure 1 shows crystallographic details of the copper single crystal which pertain to the present study. Copper has a face-centered cubic structure, as indicated, and the 110 and 100 planes are identified. It may be seen that the population density of atoms is significantly different on the two crystal planes under consideration. The dimensions for the lattice are given by Seitz (*24*) and others.

Though the exact mechanism by which the preferential wetting occurs is not understood, there is some evidence to support the hypothesis that a monolayer of cuprous oxide is adsorbed at the copper surface, after which an oriented layer of stearic acid is formed. It is further postulated that stearic acid will not wet the oriented layer of itself. Since the 100 planes are more active than 110 planes for oxidation, and since 110 planes are etched more by stearic acid than the other planes, it appears that both facts suggest a breakdown of the cuprous-oxide–stearic-acid layer at the 110 planes to give wetting. Evidence that the monolayer of stearic acid may exist has been reported by Brummage (*25*). Polar liquids, which are unable to wet their own adsorbed films, have been called *autophobic*, and a number have already been identified by Hare and Zisman (*26*).

Whatever the mechanism is, it seems reasonable that the effect can exist at a polycrystalline surface, wherein *degree of wetting* becomes a highly localized concept with each crystal in the structure having its own degree of wetting. Indeed this may be the essence of *vaporization nuclei* or *active centers* in the boiling process.

PREPARATION AND PRELIMINARY STUDIES OF COPPER SURFACES

Simultaneous with the design and construction of the system to study effects on boiling heat transfer, studies were initiated on methods of preparing the surfaces to be used in the boiling experiments. Two cylindrical copper single crystals, 1-in. in diameter and 1-in. long, were purchased for these studies. They were oriented so that one had the flat ends parallel to the 110 planes and the other had flat ends parallel to the 100 planes. For purpose of identification, they will be referred to as the 110 crystal and the 100 crystal. Both of the single crystals were mechanically polished with abrasive papers down to 0000 grit size and, then electropolished and microphotographed.

An article by Jacquet (*27*) concerning principles involved in electrolytic polishing of copper in phosphoric acid was very helpful in formulating a polishing procedure for the present studies.

Gwathmey, *et al.* (*14*), had observed that a conditioning process was required before the preferential wetting could be observed. This conditioning consisted of alternately exposing the surface to hot stearic acid and air. For this purpose, a mechanical dunking device was arranged so that the cylinders might be alternately immersed at a frequency of 20 cycles/min. The temperature of the stearic acid bath was kept at about 170°C. during the dunking operation.

Before the initial immersion period was started, periodically during the dunking, and after completion of the dunking, observations were made of contact angles between a drop of molten stearic acid and the benzene-washed top surface of each of the cylinders. The cylinders were placed on a hot plate and maintained at a temperature of about 100°C. A micrograph was prepared that showed each drop on each surface in profile so that the contact angles might be measured. Differences between the contact angles on the clean surfaces were very slight, although, before completion of the dunking, it was noted that the 100 crystal no longer appeared to be wetted during the immersion cycle. After the crystal was withdrawn from the acid, the acid would draw up and mostly drain off before the cylinder was again immersed. This was a distinct change, and neither of the other cylinders gave this appearance.

One explanation of these observations was that the conditions under which the micrographs were made were such that a film of cuprous oxide was formed on the surfaces soon after their removal from the hot stearic acid. Even if some means were used to prevent this oxidation before the stearic acid was cleaned off, the oxide layer would be formed when the cylinders were heated to 100°C. before the micrographs were taken. Thus the micrographs were in each case a measure of the contact angle of molten stearic acid with a film of cuprous oxide rather than the particular crystal face of the copper. Another possible explanation is that nonwetting develops during the formation of an oriented monolayer of the molecules. When the surfaces were washed with benzene prior to contact-angle measurements, the monolayer was removed, and the wetted condition returned.

Kruger (*28*), in discussing the work of Gwathmey *et al.*, pointed out that they were able to observe only the preferential wetting at temperatures between 160° to 270°C. Perhaps that is an explanation for the lack of difference in contact angles at 100°C., but Kruger made contact-angle measurements at 200°C. and also found only slight differences between the 110 and 100 faces. There may be a connection between the observations of Gwathmey and tests by Winkler and Vandenburg (*13*) on *wetting temperatures* of molten sodium on various surfaces.

Optical micrographs of the faces of the various cylinders were prepared, and considerable change in appearance was noted when they were compared with freshly polished surfaces. There seemed to be a growth of size in the small irregularities on the surface.

DESCRIPTION OF APPARATUS

Figure 2 is a layout of the entire boiler and vacuum system. The vacuum system was provided so as to enable operation at pressures below atmospheric. The main objective in reducing the pressure was to reduce the saturation temperature of the stearic acid and thereby reduce decomposition of the acid. Markley (*29*) states that heating stearic acid for 6 hr. at 300°C. in the presence of the oxides of copper produces not more than 6 or 7% ketones. Operating at considerably lower temperature, it is believed, minimized such decomposition.

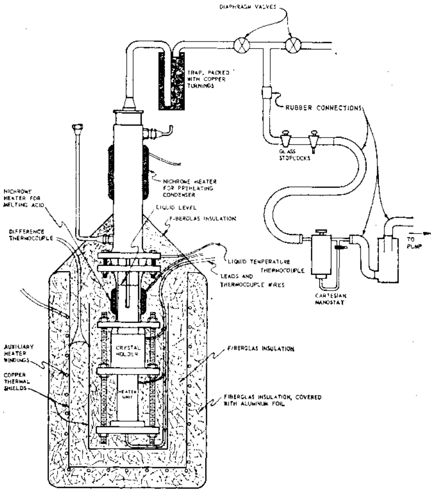

Fig. 2. Boiler and vacuum system.

The *condenser* section was an assembly of two concentric tubes, with boiling water in the annular space serving as an isothermal heat sink. The water vapor generated during experimental runs was vented to the atmosphere, and a small tube was provided for replacing water in the annulus as needed. The condenser was mounted vertically above, and concentric with, the boiler, into which the stearic acid condensate was permitted to return continuously.

The *boiler* was a 6-in. length of 1-in. Pyrex pipe surrounded with a Nichrome resistance-wire heater to melt the acid prior to boiling tests. The space in the boiler was equipped with a thermocouple well for liquid temperature measurement. The boiler was filled with liquid to within about 1-in. from the top during operation of the system.

The crystals and a polycrystalline sample used in the tests were 1 in. in diameter and 1 in. long. The crystal holder was constructed of brass, and a seal between the boiler and crystal holder was provided by a Teflon gasket. Leakage around the crystal itself was prevented by compressed asbestos packing. The holder and boiler were connected with tie bolts through flanges. The same tie bolts were extended through another flange which pushed the heater against the bottom face of the crystal in order to make good contact. The heater was made of a solid copper core, insulated with a coating of Sauereizen cement and wound with a Nichrome resistance-wire heater. Radiation shields were provided, and the power input was controlled by a 0 to 270-volt variable transformer and measured by a Weston Model 310 precision wattmeter.

The entire heater and boiler assembly was surrounded with a double-jacketed thermal shield, as shown in Figure 2.

Temperatures were measured within each crystal and in the boiling liquid by means of No. 30 copper-constantan duplex thermocouples in connection with a Leeds and Northrup Type K-2 potentiometer and an optical beam-type galvanometer system. The crystal thermocouple was located in a thin protection tube inserted in a radial hole parallel to and about ⅛ in. below the heat transfer surface. Corrections were made for the temperature drop between this thermocouple location and the heat transfer surface. The sensitivity of the temperature-measuring circuits was about 0.1°F., and the accuracy was believed to be within 0.5°F. All thermocouples were calibrated in comparison with a standard platinum, platinum-rhodium junction. The reference junction was maintained at the ice point, 32°F., in a Dewar flask.

EXPERIMENTAL PROCEDURE

Considerable effort was invested in the preparation of the sample and the system prior to operation. The system was first cleansed by boiling ethanol. The two single crystals and the polycrystal were mechanically polished and then electropolished, as described previously. All three samples were dunked simultaneously in stearic acid until preferential wetting was observed, after which the samples were left submerged until stearic acid solidified on the prepared surfaces. The sample with a protective layer of solid stearic acid was then installed in the crystal holder, which was then connected to the heater, and a thermocouple was passed into the center of the crystal. With the Teflon gasket in place, the crystal holder and heater assembly were connected to the Pyrex-pipe boiler section.

The boiler was next filled with stearic acid until the liquid was about 1 in. from the top of the tube (giving about 5 in. of liquid above the crystal surface). When this operation was completed, the boiler was connected to the condenser and the system was checked for leaks. The thermal shields were raised into position around the heater assembly, and the heater and boiler were further insulated with Fiberglas blanket and aluminum foil.

The test procedure involved the following steps:

1. The pressure was reduced to the desired level by means of the vacuum system and controlled by the Cartesian manostat.
2. The condenser section was preheated until steam was evolved from the jacket outlet.
3. The voltage on the heater was adjusted on the variable transformer so as to deliver the desired power (observed on the wattmeter).
4. After attainment of equilibrium, data were taken.

The data consisted of measured values of

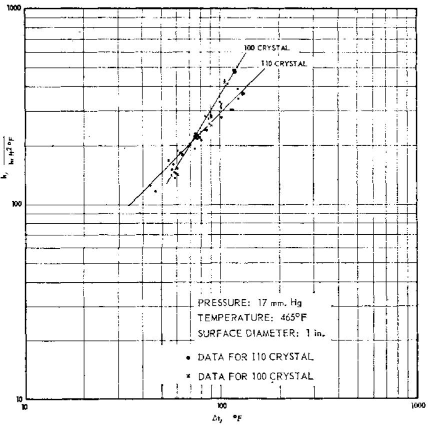

Fig. 3. Data for boiling stearic acid on copper single crystals.

power input, crystal temperature, and liquid temperature.

Data for boiling stearic acid at 17 mm. pressure are plotted on Figure 3 and summarized on Table 1.*

DISCUSSION OF RESULTS

Though at first glance it seems strange that the results from the two crystals cross as shown on Figure 3, this crossing may be explained in the following manner. At low values of heat flux, the heat exchange mechanism may be regarded as a simultaneous large contribution from nonboiling natural convection and, if there is boiling, a small contribution from the bubble formation. If the process is predominantly nonboiling natural convection, one would expect that the nonwetting interface would represent a larger thermal resistance than the wetted interface and that temperature difference would be higher for the nonwetted than for the wetted interface at the same value of heat flux. This idea is consistent with the results of the experimental studies, wherein the 100 crystal was not wetted as well as the 110 crystal and the temperature differences for the low range of heat flux were higher for the 100 crystal than for the 110 crystal.

As the heat flux increases, the rate of bubble formation increases, and the relative contribution of nonboiling natural convection is reduced. Hence, the influence of degree of wetting on ease of bubble formation assumes a more important role as heat flux increases in the nucleate boiling region. As mentioned previously, Larson (*19*), has proposed that the least wetted regions of a surface should ebullate first. This notion may be extended to postulate that the nonwetted surface requires less temperature difference than the wetted surface for the same heat flux. The data on Figure 3 tend to confirm this line of reasoning. To extend his reasoning beyond the range of the present data, one can imagine the curve for the 110 crystal proceeding to a higher maximum flux and corresponding critical temperature difference than the curve for the 100 crystal. It is hoped that this can be confirmed by subsequent experimentation.

CONCLUSIONS AND RECOMMENDATIONS

It is concluded that nonwetting decreases the heat transfer coefficient in nonboiling natural convection but increases the heat transfer coefficient in nucleate boiling. It is further concluded that contact angles are not suitable criteria for establishing degree of wetting at least with respest to heat transfer studies; that is, surface conditions under which contact angles are measured do not necessarily reproduce the surface conditions existing during a heat transfer process. This conclusion results from the fact that no differences in contact angles were observed on clean surfaces, though the different nature of interaction between the solid and liquid was apparent from the manner in which liquid drained from the surfaces and the differences in heat transfer results.

It is recommended that additional effort be expended in developing systems for similar studies but involving liquid-solid combinations other than stearic acid and copper. There is still much need for an established theory of wetting as well as a method of measuring a meaningful *degree of wetting.*

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