INVESTIGATION INTO THE POOL-BOILING CHARACTERISTICS OF GOLD NANOFLUIDS

A Thesis presented to the Faculty of the Graduate School
University of Missouri-Columbia

In Partial Fulfillment
Of the Requirements for the Degree
Master of Science

by

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AUGUST 2007
The undersigned, appointed by the Dean of the Graduate School, have examined the thesis entitled.

INVESTIGATION INTO THE POOL-BOILING CHARACTERISTICS OF GOLD NANOFLOUIDS

Presented by Jenny Ellen Jackson

A candidate for the degree of Master of Science

And hereby certify that in their opinion it is worthy of acceptance.

James Bryan

Hongbin Ma

Kattesh Katti
Acknowledgements

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Abstract

As the technological trend of increasing speed and size reduction of components continues the ability to remove high heat fluxes is becoming an ever more critical area of research. Among the topics of research in this area, the use of nanofluids as a heat transfer fluid has drawn much attention recently. Within the last five years work investigating the pool-boiling of these fluid has increased dramatically. However, the existing literature on the subject shows some varying results that need further investigation to interpret. An experimental study was performed studying the behavior of gold nanofluids during pool-boiling. In these experiments it was found that while the heat transfer decreased about 20% over the critical heat flux (CHF) was increased 2.8 times for the 3 nm sized particles. For 15 nm particles the CHF was increased over 3.5 times over pure water with a maximum at nearly 5 times amplification. After performing the experiments, scanning electron microscope images were taken of the surface as a deposition of nanoparticles was observed on the surface. It is this surface deposition that is the source of the altered boiling curves as pure water on the nanoparticle deposited surface behaves similarly to the nanofluid curve. Also, a set of experiments were also performed using HFE 7000 where no CHF enhancement was observed. Further investigation was performed to determine the source of this enhancement including an examination of the wetting characteristics of the surface. However, using existing correlations predicting the critical heat flux it was seen that the wetting of the surface likely is not the sole source of the enhanced CHF.
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<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$g$</td>
<td>Acceleration due to Gravity</td>
</tr>
<tr>
<td>$h$</td>
<td>Heat Transfer Coefficient</td>
</tr>
<tr>
<td>$h_{lv}$</td>
<td>Latent Heat of Vaporization</td>
</tr>
<tr>
<td>$q''$</td>
<td>Heat Flux</td>
</tr>
<tr>
<td>$q''_{max}$</td>
<td>Critical Heat Flux (CHF)</td>
</tr>
<tr>
<td>$q''_{max,Z}$</td>
<td>Zuber’s Prediction of Critical Heat Flux</td>
</tr>
<tr>
<td>$R_a$</td>
<td>Average Surface Roughness</td>
</tr>
<tr>
<td>$R_q$</td>
<td>RMS Surface Roughness</td>
</tr>
<tr>
<td>$\beta_r$</td>
<td>Receding Contact Angle</td>
</tr>
<tr>
<td>$\lambda_D$</td>
<td>Helmholtz Wavelength</td>
</tr>
<tr>
<td>$\rho_l$</td>
<td>Liquid Density</td>
</tr>
<tr>
<td>$\rho_v$</td>
<td>Vapor Density</td>
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<tr>
<td>$\sigma$</td>
<td>Surface Tension</td>
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1. Introduction

As the technological trend of increasing speed and size reduction of components continues the ability to remove high heat fluxes is becoming an ever more critical area of research. In an attempt to resolve these issues several methods are being investigated. One area that has drawn considerable attention in recent years has been the use of nanofluids as a heat transfer fluid. The idea of using these nanofluids, defined as a solution with particles less than 50 nm in size, was first introduced by Choi [1] in 1995. Since then interest in nanofluids has grown exponentially. His and later research by Das et al. [2] and Eastman et al. [3] found that with nanoparticles suspended in fluids, the effective thermal conductivity could be increased.

While much of the previous work investigating the use of nanofluids as a heat transfer fluid has focused on the single phase aspects such as thermal conductivity and convection, within the last five years groups have begun exploring the characteristics of boiling nanofluids. When considering the effect of nanofluids on the boiling traits unique aspects come to mind such as the behavior of the nanoparticles as the fluid transitions between phases. A summary of the existing work in the field including experimental setup and results is shown in Table 2. The existing literature on the subject shows some varying results that need further investigation to interpret.
Fig. 1. Selected experimental results from reported boiling experiments. Empty points denote baseline water and solid points denote nanofluid data.
Table 1. Characterization of nanofluids used in reported experiments.

<table>
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<tr>
<th>Reference</th>
<th>Nanofluid</th>
<th>Particle Size</th>
</tr>
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<tr>
<td>[4]Das</td>
<td>1, 2, 4 % vol. Al₂O₃ in DI water</td>
<td>12-270 nm 58.4 nm avg.</td>
</tr>
<tr>
<td>[5]Bang and Chang</td>
<td>0.5, 1, 2, 4 % vol. Al₂O₃ in DI water</td>
<td>10-100 nm 47 nm avg.</td>
</tr>
<tr>
<td>[6]Wen and Ding</td>
<td>0.08, 0.18, 0.24, 0.31 % vol. Al₂O₃ in DI water electrostatically stabilized</td>
<td>10-50 nm</td>
</tr>
<tr>
<td>[7]Vassallo</td>
<td>0.5 % vol. silica nanoparticles in DI water</td>
<td>15 and 50 nm nominal</td>
</tr>
<tr>
<td>[8]You</td>
<td>0.000025, 0.00013, 0.00025, 0.00063, 0.0013 % vol. Al₂O₃ in DI water</td>
<td>Not stated</td>
</tr>
<tr>
<td>[9]Das</td>
<td>0.1, 1, 2, 4 % vol. Al₂O₃ in DI water</td>
<td>20-50 nm 38 nm avg.</td>
</tr>
<tr>
<td>[10]Kim</td>
<td>10⁻⁵ to 0.1 % vol. TiO₂ in water</td>
<td>10-500 nm 85 nm avg.</td>
</tr>
<tr>
<td>[11]Wen</td>
<td>0.31, 0.44, 0.57, 0.72 % vol. TiO₂ in water, electrostatically stabilized</td>
<td>50-400 nm Peak at 169 nm</td>
</tr>
<tr>
<td>Reference</td>
<td>Test Setup</td>
<td>Pressure</td>
</tr>
<tr>
<td>------------</td>
<td>----------------------------------------------------------------------------</td>
<td>----------</td>
</tr>
<tr>
<td>[4]Das</td>
<td>Tube heaters of 4, 6.5, 20 mm diameter. Typical surface roughness: 0.37-0.45 μm</td>
<td>1 atm</td>
</tr>
<tr>
<td>[5]Bang and Chang</td>
<td>Vertical and horizontal Heating Section Heater 4 mm x 100 mm</td>
<td>1 atm</td>
</tr>
<tr>
<td>[6]Wen and Ding</td>
<td>150 mm diameter horizontal heater</td>
<td>1 atm</td>
</tr>
<tr>
<td>[7]Vassallo</td>
<td>0.4 mm diameter NiCr wire</td>
<td>1 atm</td>
</tr>
<tr>
<td>[8]You</td>
<td>Flat square heater (1x1 cm²)</td>
<td>19.9 kPa</td>
</tr>
<tr>
<td>[9]Das</td>
<td>Cartridge heater 20 mm diameter Average initial surface roughness: Ra=0.387 and 1.15 μm Rq= 0.458 and 1.54 μm</td>
<td>1 atm</td>
</tr>
<tr>
<td>[10]Kim</td>
<td>NiCr wire 0.2 mm diameter Investigation on CHF</td>
<td>1 atm</td>
</tr>
<tr>
<td>[11]Wen</td>
<td>150 mm diameter polished stainless steel disc</td>
<td>1 atm</td>
</tr>
</tbody>
</table>

One aspect of these experiments that is important to compare is the different types of fluids used in the experiments. Of the groups compared, all used water as the base...
fluid for the particles. However the type of particles used varies. Many of the reported
groups performed their experiments using alumina nanoparticles while other particles
cited are silica and TiO$_2$. Even among the alumina groups the results vary considerably.
Das et al. [9] and Bang and Chang [5] show similar results for their experiments as can be
seen in Fig. 1. However, while these groups indicate a decrease in the heat transfer, You
et al. [8] shows no change in the heat transfer coefficient and Wen and Ding [6]
demonstrate an improvement of the boiling heat transfer. Also, while both Bang and
Chang and You saw an increase in the critical heat flux, the amount of increase between
the groups was considerable 32% compared to 200%. From these results it seems that the
type of nanoparticle is not the only factor affecting the outcomes of these experiments.
Another aspect of the fluid that should be considered is how the fluid is created. Many
times stabilization agents are added to the fluid which may act as a surfactant changing
the boiling properties. If this were the case it may be difficult to determine the source of
any changes seen between pure water and the nanofluid. Wen and Ding used an
electrostatic stabilization method in which the pH value of the suspension is adjusted
away from the iso-electrical point of alumina as to take advantage of the repulsive forces
due electric double layer surrounding the particles. It may be this change in the nanofluid
characteristics which accounts for the significantly different results they obtained.
However, whether the source is the change in the fluid properties or merely the
improvement of the particle suspension cannot be determined.

The nanofluid concentration is also a factor that deserves consideration. Many
of the reported groups have investigated the effect of various suspension concentrations.
Das et al. reported a decrease in the heat transfer coefficient with an increase in the
nanofluid concentration. The other groups focused more on the CHF of the fluids and found that as concentration increases the CHF increases. It should also be noted that the concentrations used by You et al. and Kim et al. are considerably smaller than those used by the other groups, however both groups saw a 3 times increase in the CHF compared to the 30% and 60% increases seen by Bang and Chang and Vassallo et al., respectively.

Another aspect of the experiments presented needing consideration is the heating surface. First, the type surface used will affect the outcome of the experiments. Vassallo and Kim’s NiCr wire surfaces will behave differently to the round heaters used by Das and Wen and Ding and the flat surfaces used by Bang and Chang and You. Beyond the macroscopic aspects of the surface the microscopic properties such as the roughness of the surface will also affect the boiling curves. Das et al. compared two different surface roughnesses and found that a rougher surface aids the heat transfer. Ferjancic and Golobic [12] investigated the effect of surface condition on the CHF of pure water and observed an increase of the CHF up to 20% as the surface roughness increases. They also asserted a 50% increase when an etched surface is used compared to a ground surface of the same roughness. Of the sources cited, only Das and Bang and Chang comment on the condition of their surfaces prior to boiling presenting values for the typical surface roughness of the heater surface. These groups also noticed a change in the measured surface roughness after boiling the nanofluids due to surface deposition presenting another issue to address. Das et al. saw a decrease in the measured roughness while Bang and Chang observed an increase. The difference is likely due to the fact that Das’s initial surface roughness was larger than the measured particle size and Bang’s initial roughness was smaller that the measured particle size. Vassallo and Kim et al. also noticed a
coating on the wire after testing. These results point to surface deposition affecting the boiling characteristics of the nanofluids. Kim et al. and Bang and Chang noted that with increasing concentration surface deposition increased.

Finally, if there is an issue with the settling of the nanoparticles and/or a surface deposition during the boiling experiments the experimental procedure will also play a significant role in the outcome. Mainly, the time the fluid is in the test set up and the number of trials is important. Few of the existing sources investigating the pool-boiling of nanofluids address this issue. Das et al. as well as Wen and Ding cleaned the surface between each trial of their reported experiments removing any deposition that may have occurred. Das et al. did state however that fouling of the surface did occur during trials taken after four days and two weeks resulting in a decrease in the heat transfer coefficient. It is also unclear in many of the sources when the presented trials were performed. For example was it the first trial, a trial recorded after several runs, or an average of several trials performed.

Recently work has been performed to find the mechanism by which nanofluids create this dramatic increase in the critical heat flux. In experiments performed by Kim et al. [13] the contact angle of both water and nanofluids on water boiled surface and a nanofluid boiled surface were measured. Their results show that while the water and the nanofluids perform similarly on the surfaces, the contact angle on the clean surface is about 70° compared to 20° on the Al₂O₃ nanoparticle deposited surface.

The investigation presented here investigates the pool-boiling characteristics of gold nanofluids in water. The objectives of this study were to perform a set of reliable, well-documented and repeatable experiments, examine the boiling characteristics such as
heat transfer coefficient and critical heat flux (CHF), and then attempt to resolve the source of any differences observed by adding the nanoparticles.

2. Experimental Setup and Design

2.1. Nanofluid Characterization

The experiments were conducted with water and gold nanofluid generated via the citrate reduction synthesis process and has a concentration of approximately 0.0003% vol. (0.058 grams per liter). This nanofluid was chosen due to its well established manufacturing method, the fact that gold is a noble metal, the ability particles to maintain suspension, and its ability to create uniform nanoparticles for various sizes. Particle sizes of nominally 3 nm and 15 nm were used. Transmission electron microscope (TEM) images of the gold nanoparticles are shown in Fig. 2. From these images the particle size distribution the nanofluids were determined and are shown in Fig. 3. The 3 nm nominal nanoparticle size averaged 4.5 nm with a standard deviation of 1.5 nm while the 15 nm nominal nanoparticle averaged 16.8 nm with a standard deviation of 2.7 nm.

Fig. 2. TEM image of the a) 3 nm and b) 15 nm gold nanoparticles.
Fig. 3. Particle size distribution of 3 nm and 15 nm nanofluids.

2.2. Experimental Setup

The experimental system is comprised of a nucleate boiling chamber, a data acquisition system, a power supply and a circulating water bath. The pool boiling chamber consists of a sealed cylindrical container with a copper condensing coil at the top, a horizontal copper boiling surface at the bottom, and several temperature sensors (Fig. 4). The chamber is constructed from acrylic to allow for visual observation of the boiling process. It has an internal diameter of 8.9 cm and a height of 25.4 cm. A Teflon insert in the bottom of the acrylic container supports and insulates the coupon. An Ultem flange is used to hold the coupon in place. The pool boiling chamber is sealed with o-rings for sub-atmospheric pressure experiments. An Omega OVG64 digital vacuum gauge is used as a reference to make certain that a constant pressure is maintained throughout each trial. All tests were conducted with boiling occurring at room temperature thereby eliminating the need for insulation around the acrylic. This corresponds to an equilibrium pressure inside of the pool boiling chamber of roughly 2.3 kPa. Room temperature vulcanization (RTV) silicone was used between the heater and
the Ultem flange to prevent bubble nucleation from the side of the heated surface. Two RTDs were placed in the boiling chamber, one in the bulk fluid and one in the vapor space above the fluid. A Keithley Integra series - 2701 data acquisition system recorded the temperature and heater voltage information. Water was pumped through the condensing coil by a Neslab RTE-111 circulator to maintain a constant vapor temperature in the boiling chamber.

Fig. 4. Schematic and images of the pool boiling chamber (A and C) and the copper coupon (B), where 1) vapor RTD, 2) liquid RTD, 3) Teflon base, 4) condenser coil, 5) acrylic cylinder, 6) copper coupon, and 7) Ultem flange. All dimensions in cm.

The cylindrical coupon is made of 99.999% pure copper and provides a 1 cm$^2$ boiling surface. A small resistance heater was brazed to the bottom surface of the coupon and two T-type thermocouples were inserted into 0.75 mm holes drilled into the coupon (Fig. 4B) to measure the temperatures near the surface of the coupon and in the middle of the coupon. Two heating surface preparations were used for the experiments. The first surface was the machined finish created by the milling of the coupon. Scanning Electron
Microscope (SEM) images of this surface are shown in Fig. 5. The surface roughness values $R_a$ and $R_q$ were measured using a profilometer and found to average to 584.2 nm and 711.2 nm, respectively for this surface. The second surface condition was a polished surface achieved by polishing the coupon with 1200 grit emery paper. SEM images of the polished surface are shown in Fig. 6. The resultant average surface roughness, $R_a$, and RMS roughness, $R_q$, measured for the polished surface were 177.8 nm and 254.0 nm, respectively.

Fig. 5. SEM images of the machined heating surface before boiling at a) 400x and b) 2000x magnification.
2.3. Experimental Procedure

In order to determine the extent of degradation to the heating surface, the coupon was first examined using a scanning electron microscope (SEM), shown in Fig. 6. Once inspected, the heating surface was inserted into the test section previously described. A trial of pure water was run before each set of nanofluid trials to ensure consistent performance of the surface. In order to accurately gauge heat input the resistance of the heater was recorded. A vacuum was pulled on the air tight test section and the appropriate fluid was drawn into the chamber. Once the proper amount of fluid was inserted, a vacuum was pulled once again in order to both reduce the pressure in the chamber and remove any non-condensable gases. Both the vacuum and charging ports were sealed and the recirculating chiller was set to 19 °C. Next, 16 watts were applied to
the heater for 30 minutes and then the system was degassed. The power source was shut off and the system was allowed to return to ambient steady state conditions.

The procedure executed for each trial involves varying the heat applied to the copper coupon. The heat flux was varied from 0 kW/m$^2$ up to the critical heat flux ensuring multiple data points in each boiling regime. At each point along the curve, the temperatures were monitored to ensure the system achieved steady-state. Once steady state was reached, 100 measurements, taken over approximately three minutes, were recorded. The procedure was repeated without removing the fluid from the chamber until the boiling curve stabilized and produced repeatable results. Once repeatable data was achieved, the coupon was then carefully removed and placed in a sealed dry nitrogen gas environment to prevent further degradation to the surface during idle time and transportation to the SEM facility. The tested surface was then viewed by the SEM for comparison with the pre-tested surface.

2.4. Data Reduction and Measurement Uncertainty

To attain the data for each point for each trial the 100 recorded measurements were averaged. The final data is an average of the data taken over three trials performed on multiple days. The surface temperature was calculated using Fourier’s law with the heat flux and the temperature at thermocouple location one. The superheat is then determined as the difference between the surface temperature and the bulk liquid temperature. The heat flux applied to the system was determined using the electrical power applied to the heater. The heat transfer coefficient is computed as the heat flux over the superheat.
The uncertainty for the specified experiment was determined using standard error analysis methods with calibration from a Kaye standard RTD temperature probe. Uncertainty in the measurements was determined for the thermocouples, the RTDs, and the input voltage. Calculated uncertainties include wall superheat, wall temperature, heat flux, and heat transfer coefficient. The maximum and minimum uncertainties for both measured and calculated quantities are shown in Table 3.

<table>
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<tr>
<th>RTD_vap (°C)</th>
<th>RTD_liq (°C)</th>
<th>T1 (°C)</th>
<th>T2 (°C)</th>
<th>EMF (V)</th>
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<tr>
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<td>0.03</td>
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<td>0.08</td>
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<tr>
<td>Avg</td>
<td>0.10</td>
<td>0.09</td>
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<table>
<thead>
<tr>
<th>Tsuf (°C)</th>
<th>Tsup (°C)</th>
<th>q'' (kW/m²)</th>
<th>h (kW/m²K)</th>
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<tbody>
<tr>
<td>Max</td>
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<td>Avg</td>
<td>0.22</td>
<td>0.32</td>
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3. Results and Discussion

3.1. Experimental Results

Prior to performing the nanofluid experiments a set of experiments was performed using pure water. This ensured that the surface was acting consistently as well as provided the baseline for comparison. Knowing that the method in which the nanofluid was manufactured may affect the outcome of the experiments, a set of boiling experiments was performed using the basefluid (Solution B from the nanofluid preparation procedure in Appendix A). The results from these experiments are seen in Fig. 7.
From this graph, it is seen that for the basefluid trial the heat transfer coefficient decreases 10% for given input and the critical heat flux is 1.5 times higher. Once this base comparison was performed trials were performed with the 3 nm nanofluid. Both curves are averages of three trials take over several days.

The heat flux versus wall superheat results for the 3 nm nanofluid on the polished surface are shown in Fig. 8. The pure water curve and the settled nanofluid curve (nanofluid trials 5-7) are averages of daily trials performed at the same conditions over four days and the error bars represent the overall uncertainty of the measurements. As one can see, when the nanofluids were added, the boiling curve began to shift to the right signifying a decrease in heat transfer. For each successive trial the curve continued to shift to the right until the third trial, after which the boiling performance remained essentially constant. To determine the overall effect of the nanoparticles on heat transfer,
the heat transfer coefficient was plotted against the heat flux. The graph, shown in Fig. 9, confirms that the heat transfer coefficient decreased 20% for the 3 nm nanofluid solution over pure water. Notice, however, that in the natural convection region (up to 10 kW/m$^2$) little to no difference is seen between the nanofluids and the pure fluid. Also obvious in Fig. 8 is a significant increase in critical heat flux from pure water to the nanofluid. The first nanofluid trial increased the heat flux about 1.8 times the pure water trials. Overall for the 3nm nanofluid solution, the critical heat flux was 2.8 times higher than that of pure water. Similar plots for all nanofluid trials can be found in Appendix B.

![Graph showing heat flux versus wall superheat results for pure water and 3 nm nanofluid on polished surface at 2.3 kPa (19°C).](image)

*Fig. 8. Heat flux versus wall superheat results for pure water and 3 nm nanofluid on polished surface at 2.3 kPa (19°C).*
Fig. 9. a) Heat transfer coefficient versus heat flux results for pure water and nanofluid on polished surface at 2.3 kPa (19°C).

After the experiments were performed a thin, dark film was observed on the heated copper surface seen in Fig. 10 (center). In order to determine the nature of the film, scanning electron microscope (SEM) pictures were taken of the surface and are shown in Fig. 10. In the images taken prior to performing the experiments, lines and smears created by the polishing of the surface are visible (Fig. 6). By comparing these images to images taken after boiling, it is evident that a layer has formed on top of the copper. Energy dispersive (X-ray) spectrometry was then used to examine the composition of the surface. The exact location of the test sites and the corresponding compositions are presented in Fig. 11 and Table 4, respectively. One can see that gold is detected indicating that nanoparticles have been deposited on the surface during the boiling process. This deposition occurred despite the fact that overall the particles remained suspended. It is speculated that these particles have changed the coupon
surface altering its characteristics and causing the shifted boiling curves. It should be noted that carbon particles were also observed on the surface of the coupon. However, the carbon particles seem to make up only a small percentage of the surface.

**Fig. 10.** SEM images of the polished heating surface after boiling 3 nm nanofluid taken at a) 50x, b)250x, c)1000x, and d) 5000x.

**Fig. 11.** Image and locations used for energy dispersive (x-ray) spectrometry. Taken at 3000x magnification.
Table 4. Composition of coupon surface as determined using energy dispersive (x-ray) spectrometry.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>O</th>
<th>Cu</th>
<th>Au</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pt. 1</td>
<td>Weight %</td>
<td>9.16</td>
<td>2.77</td>
<td>8.39</td>
</tr>
<tr>
<td></td>
<td>Atom %</td>
<td>51.79</td>
<td>11.79</td>
<td>8.97</td>
</tr>
<tr>
<td>Pt. 2</td>
<td>Weight %</td>
<td>78.40</td>
<td>21.60</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td>Atom %</td>
<td>82.86</td>
<td>17.14</td>
<td>--</td>
</tr>
<tr>
<td>Pt. 3</td>
<td>Weight %</td>
<td>9.74</td>
<td>2.26</td>
<td>7.88</td>
</tr>
<tr>
<td></td>
<td>Atom %</td>
<td>54.68</td>
<td>9.54</td>
<td>8.36</td>
</tr>
</tbody>
</table>

A set of experiments was also performed with the 3 nm fluid on a machined surface. When the results for the two surfaces are compared in Fig. 12, one sees that for water the curve for the polished surface (SA) is shifted to the right of that for the machined surface (S1). This is expected due to the reduced roughness of the polished surface shown in Table 5. A smoother surface will generally have a lower nucleation site density causing this shift to the right. The 3 nm curves however follow right along each other. This is likely due to the deposition of the nanoparticles on the heating surface. As one compares the SEM images of the two surfaces, it is seen that while the machined surface is visibly rougher than the polished surface before boiling (Fig. 5 and Fig. 6), after boiling the surfaces appear somewhat similar (Fig. 13 and Fig. 10). From Table 5 it is seen that there is about a 200 nm increase in the surface roughness for both the machined and the polished surface indicating that the layer thickness for both sets of trials is likely similar. The similar film thickness is likely what caused the two trials to have similar heat transfer qualities. The difference in the CHF between the 3 nm trials may be a result of the increased surface roughness [14].
Fig. 12. Boiling curves for experiments performed with the 3 nm particles at 2.3 kPa (19°C).

Table 5. Surface roughness measurements for all trials. All units in nm.

<table>
<thead>
<tr>
<th></th>
<th>Before</th>
<th>Post 3 nm</th>
<th>Post 15 nm (1)</th>
<th>Post 15 nm (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Machine Finished Surface</strong></td>
<td>Ra</td>
<td>584.2</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>Rq</td>
<td>711.2</td>
<td>1092.2</td>
<td>N/A</td>
</tr>
<tr>
<td><strong>Emery Polished Surface</strong></td>
<td>Ra</td>
<td>177.8</td>
<td>1422.4</td>
<td>1143</td>
</tr>
<tr>
<td></td>
<td>Rq</td>
<td>254.0</td>
<td>533.4</td>
<td>2057.4</td>
</tr>
</tbody>
</table>
Once the 3 nm trials were completed, the effect of particle size was then investigated. For these trials the 15 nm nominal particle size and the polished surface were used. Three separate sets of trials were performed to examine the repeatability of the results. The results of these experiments are shown in Fig. 14 along with the pure water and 3 nm cases. From this figure, it is seen that the CHF for the 15 nm trials are 790 kW/m$^2$, 1200 kW/m$^2$, and 850 kW/m$^2$ which is nearly 5 times higher than the water case for set 2 and over 3 times higher for sets 1 and 3. The 15 nm cases are even 25% higher than the 3 nm case.
Fig. 14. Boiling curve results for 3nm and 15 nm trials at 2.3 kPa (19°C).

The source of the difference between these trials is likely due to the difference in the surface deposition created by the boiling. Pictures of the heater surface immediately after boiling the nanofluids are shown in Fig. 15. One can see the difference in the structure of the surface. The difference is further illustrated by comparing the SEM images in Fig. 16 and Fig. 17. From these images it is seen that the features created in the first set are noticeably larger and of a different nature than those created by the third set. This is confirmed by the surface roughness measurements for these surfaces presented in Table 5. Post boiling surface roughness data and SEM images are not available for the second set of trials as the heater surface was ruined during testing. The surface roughness for the first set of trials is 25% higher than that for the third set. A difference in the deposition layer thickness is likely the source of the difference in heat transfer between the 15 nm
trials. While the film thickness could not be directly determined, using the surface roughness measurements, the layer thickness has minimum of about 1.25 μm for set one and 0.95 μm for set three and a maximum for all trials of 3.75 μm assuming all of the gold was to deposit solidly on the heater surface. As the second and third sets were performed, significant accumulation of nanoparticles on the side of the testing chamber was observed as seen in Fig. 18. This resulted in the concentration of the fluid being greatly reduced as evident in images of the nanofluid before and after in Fig. 19.

![Fig. 15. Pictures of the boiling surface after boiling the 15 nm nanofluids.](image-url)
Fig. 16. SEM images of polished surface after boiling 15 nm nanofluid (1st set) at 50x, 250x, 1000x, 5000x.

Fig. 17. SEM images of polished surface after boiling 15 nm nanofluid (3rd set) at 50x, 250x, 1000x, 5000x.
Seeing this shift in the boiling curves and the dramatic increase in critical heat flux, it was then attempted to try and narrow down the source of these outcomes. The noticeable deposition of nanoparticles on the surface was suspected to be the cause of the transformation. Once the nanofluid boiling trials were completed, water was then boiled using the nanoparticle deposited surface. It was observed in these experiments that even once the nanofluid was removed the boiling curve followed right along with the curve produced with the nanofluid in the chamber maintaining its state of elevated heat flux,
seen in Fig. 20. This observation points to the altered surface characteristics being the driving force behind the enhanced CHF not the addition of nanoparticles to the boiling fluid.

![Boiling curves comparing water and nanofluids on clean and nanoparticle deposited surface at 2.3 kPa (19°C).](image)

Fig. 20. Boiling curves comparing water and nanofluids on clean and nanoparticle deposited surface at 2.3 kPa (19°C).

A set of experiments was also performed examining the boiling characteristics as the saturation pressure moves toward ambient. The boiling curves for the clean, polished surface and the nanoparticle deposited surface are shown in Fig. 21 and Fig. 22, respectively. It is seen that as the saturation pressure is increased the curve shifts to the left and the CHF increases as is expected. On the polished surface the critical heat flux increases from 250 kW/m² at a saturation temperature of 19°C (2.3 kPa) to 400 kW/m² at a saturation temperature of 75°C (38.6 kPa). For the nanoparticle surface, the increase is from 870 kW/m² at a saturation temperature of 19°C to 1100 kW/m² at a saturation
temperature of 75°C. This shows that while there is a 3.5 times increase in CHF at room temperature the increase in CHF at 75°C is only 2.75 times.

Fig. 21. Boiling curves for the polished surface under various saturation conditions.

Fig. 22. Boiling curves for the nanoparticle surface under various saturation conditions.
From images taken of the nucleation, shown in Fig. 23, the difference in the vapor density at these pressures is obvious. Plots of heat transfer coefficient versus saturation pressure are shown in Fig. 24 for the clean surface and Fig. 25 for the nanoparticle surface.

*Fig. 23. Images of boiling water on the nanoparticle surface at 200 kW/m$^2$ (left) and 500 kW/m$^2$ (right) at 2.3 kPa (top) and 38.6 kPa (bottom).*
Fig. 24. Heat transfer coefficient versus pressure under different heat loads for polished surface.

Fig. 25. Heat transfer coefficient versus pressure under different heat loads for nanoparticle surface.
To further the investigation into the nature of the surface a set of boiling experiments was performed using HFE7000 as the fluid and compared to HFE7000 boiled on the polished copper surface. HFE7000 was chosen for its wetting characteristics as it is hydrophilic by nature thus any affects created by the change in wetting should be minimized. The results of these trials are shown in Fig. 26. The most notable feature seen from this comparison is that the critical heat flux for HFE 7000 is the same on both the clean surface and the deposited surface. The Zuber prediction for HFE is 140 W/m² and the adjusted value for an infinite, flat plate is 159 W/m². It was also found that considerable historesis is seen on the nanoparticle surface while the clean surface exhibited little. It was noticed that bubble diameter for the HFE 7000 on both surfaces is considerably smaller than that of the water. The differences in the nucleation characteristics can be seen in Fig. 27 and Fig. 28.

![Boiling curves for HFE7000 at ~52 kPa (19°C).](image)

*Fig. 26. Boiling curves for HFE7000 at ~52 kPa (19°C).*
Fig. 27. Images of water boiling on nanoparticle surface at 200 kW/m$^2$ (left) and 500 kW/m$^2$ (right).

Fig. 28. Images of HFE7000 boiling on nanoparticle surface at 6.6 kW/m$^2$ (left) and 160 kW/m$^2$ (right).
Contact angle measurements were taken on the base copper surface and the gold nanoparticle deposited surface and can be seen in Fig. 29. The average contact angle for both pure water and the 15 nm nanofluid are summarized in Table 6.

![Droplet images for a) water on the polished surface b) water on the nanoparticle deposited surface c) nanofluid on the polished surface and d) nanofluid on the nanoparticle surface.](image)

**Fig. 29.** Droplet images for a) water on the polished surface b) water on the nanoparticle deposited surface c) nanofluid on the polished surface and d) nanofluid on the nanoparticle surface.

**Table 6. Average measured contact angles.**

<table>
<thead>
<tr>
<th>Surface</th>
<th>Water</th>
<th>15 nm Nanofluid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean Copper Surface</td>
<td>116±9</td>
<td>114±3</td>
</tr>
<tr>
<td>Nanoparticle Deposited Surface</td>
<td>158±2</td>
<td>158±2</td>
</tr>
</tbody>
</table>

Like Kim [13] it is seen that the pure water and the nanofluid perform similarly. However, the contact angle of the nanoparticle deposited surface is considerably larger than that on the clean copper surface. This increase in contact angle is the opposite of the results shown in Kim where they show a decrease from 70° to 20°. The difference in the results may be due to the fact that Kim was investigating oxide nanoparticles where the data presented here is using gold nanoparticles. An oxide surface will have improved...
wetting over the non-oxidized surface. However, the high surface energy of pure gold would typically create a wetting surface which is again not what was seen. The structure of the nanoparticle-deposited surface is likely the source for this increase in apparent contact angle.

### 3.2. Model Comparison

When one looks at the work performed attempting to predict CHF, the most widely used correlation is the hydrodynamic model determined by Zuber [15]. The correlation predicts the critical heat flux as

\[
q_{\text{max},Z}^* = \frac{\pi}{24} \rho_v h_f \left( \frac{g \sigma (\rho_l - \rho_v)}{\rho_v^2} \right)^{1/4}.
\]

(1)

However, this model does not take into account surface affects such as geometry, surface condition, and wettability. Since this model was established, several groups have created additions to the correlation to adjust for the geometry of the heater surface [16]. Using Eq. 1 the Zuber prediction of CHF of water at 20°C was found to be 218 kW/m². From Fig. 2 one can see that this is within 15% of the value obtained on the clean copper surface. If one uses the adjustment factor on Zuber’s equation of 1.14 presented by Lienhard and Dhir [16] for an infinite (30 times the capillary length scale), flat surface, it predicts the critical heat flux at 248 kW/m² which is right at the value for water on the clean surface. However, once the nanoparticles have deposited on the surface a critical heat flux is achieved of 3.5 times the predicted value. This enhancement must be a result of the surface condition. However, little work has been performed looking at the effects of the surface condition or wettability. Ramilison et al. [14] generated a correlation of
the critical heat flux that attempts to capture the effect of surface conditions such as surface roughness and contact angle. Their correlation follows the equation

\[
\frac{q_{\text{max}}}{q_{\text{max},Z}} = 0.0336(\pi - \beta_r)^{0.125} R_q^{0.3} 
\]

(2)

where \(\beta_r\) is the receding contact angle in radians and \(R_q\) is the RMS roughness parameter. The estimate of the roughness parameter used to create this correlation range from 2 \(\mu\)m for a surface mirror polished with alumina to 12 \(\mu\)m for a surface finished with #60 emery paper. The requirements for the boiling experiment to be used for this correlation include having vertical side wall surrounding the surface and the heater must have a characteristic length more that twice the Helmholtz wavelength

\[
\lambda_{\text{hp}} = \left[ \frac{3\sigma}{(\rho_l - \rho_r)g} \right]^{1/2} . 
\]

(3)

Kandlikar [17] developed the correlation

\[
\frac{q_{\text{max}}}{q_{\text{max},Z}} = \frac{24}{\pi} \left( \frac{1 + \cos \beta_r}{16} \right) \left[ \frac{2}{\pi} + \frac{\pi}{4} \left( 1 + \cos \beta_r \right) \right]^{1/2} 
\]

(4)

to account for the surface wetting affect on the critical heat flux for a horizontal surface. For this correlation a contact angle for a water-copper system was assumed to be 45°.

Using the RMS surface roughnesses shown in Table 5 and the contact angle measurements listed in Table 6, both the Ramilison and Kandlikar correlations predict values considerably lower than Zuber’s prediction and the results presented above (shown in Table 7). However, these contact angle measurements are equilibrium contact angles while both correlations use the dynamic receding contact angles in their correlations. While the receding contact angles for our surfaces were not measured, it is known that the receding contact angle would be less than the equilibrium contact angle.
increasing the predicted value. If one uses the value of 45° for the receding contact angle of water on copper that Kandlikar assumes, the Ramilison correlation still predicts the CHF to be much lower than the Zuber prediction but the Kandlikar correlation predicts the CHF to be 1.15 times the Zuber prediction which is very close to our experimental results. By increasing this receding contact angle by 36% as was seen for the static contact angle, a receding contact angle for the structured surface of 61.3° was determined which led to a 17% decrease in the CHF prediction calculated using Kandlikar. However if a 71% decrease in the contact angle occurred as was seen by Kim, the Ramilison correlation predicts a value nearly twice the polished surface yet still lower than the Zuber correlation and Kandlikar predicts a 22% increase. While both correlations predict an increase in the critical heat flux with improved wetting, neither predicts an increase of 3.5 times as was seen experimentally. Therefore, even if the increase in apparent contact angle was only due to the structure of the surface and a decrease in the actual contact angle similar in magnitude to that seen by Kim, the entire CHF improvement seen is not accounted for by the wetting characteristics.

Table 7. Correlation predictions of CHF.

<table>
<thead>
<tr>
<th>$R_q$</th>
<th>$\beta_r$</th>
<th>$q''<em>{\text{max}}/q''</em>{\text{max,Z}}$</th>
<th>Experimental</th>
<th>Ramilison</th>
<th>Kandlikar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polished</td>
<td>0.3</td>
<td>116</td>
<td>1.1</td>
<td>0.38</td>
<td>0.28</td>
</tr>
<tr>
<td>Structured</td>
<td>1.6</td>
<td>158</td>
<td>3.9</td>
<td>0.002</td>
<td>0.03</td>
</tr>
<tr>
<td>Polished</td>
<td>0.3</td>
<td>45</td>
<td>1.1</td>
<td>0.47</td>
<td>1.15</td>
</tr>
<tr>
<td>Structured</td>
<td>1.6</td>
<td>613</td>
<td>3.9</td>
<td>0.32</td>
<td>0.95</td>
</tr>
<tr>
<td>Structured</td>
<td>1.6</td>
<td>12.7</td>
<td>-</td>
<td>0.89</td>
<td>1.40</td>
</tr>
</tbody>
</table>
4. Conclusion

A set of experiments was performed examining the pool-boiling characteristics of nanofluids. It was found from these experiments that while the heat transfer coefficient decreased significantly the maximum attainable heat flux was dramatically increased. The CHF attained using 3 nm nanofluids was 2.8 times the CHF of water on the clean surface and using 15 nm nanofluids was up to 4.8 times the CHF of water on the clean surface. However, a thin film was observed on the heating surface after boiling and by examining SEM images of the surface and using x-ray spectrometry it was determined that this film was deposited nanoparticles. It was then confirmed that it was this surface deposition that resulted in the adjusted boiling curves and enhanced CHF as pure water boiled on this surface performed similarly to the nanofluids. An investigation into the aspect of the surface that improved the CHF looked at its wetting characteristics. Contact angle was examined; however, the nanoparticle surface actually produced a higher apparent contact angle than the clean, polished surface. An increase in contact angle would actually serve to decrease the critical heat flux. A second look at wetting’s effect on the CHF was performed by running a set of experiments using the highly wetting HFE7000 were then performed. From these tests it was found that there was no CHF enhancement when boiling HFE7000. By looking at the nucleation characteristics, it is seen that the HFE7000 has a significantly smaller bubble size than the water and that little difference is seen between the surfaces for HFE7000. It is speculated that the nanoparticle surface serves to reduce the nucleation size and increase site density which may not be seen for the HFE7000 due to its already small characteristic length.
From this study it was seen that a significant enhancement in the critical heat flux can be attained by creating a structured surface from the deposition of nanofluids. If the deposition film characteristics such as thickness and the structure can be controlled it may be possible to increase the CHF with little reduction in the heat transfer. While the nanoparticles themselves cause little to no difference in the pool-boiling characteristics of water, the boiling of nanofluids shows promise as a simple way to create an enhanced surface.
References


Appendix A

Citrate reduction gold nanofluid preparation method.

β Create the following solutions

1. 1% w/v Tannic acid (0.1 g per 10 ml)
2. 1% w/v Trisodium citrate-2H₂O (0.1 g per 10 ml)
3. 25 mM K₂CO₃ (0.035 g per 10 ml)
4. 1% HAuCl₄ (0.1 g per 8.5 ml - compensating for hydration)

β Combine these solutions into two solutions as follows for desired particle size

<table>
<thead>
<tr>
<th>Solution Size</th>
<th>Particle Size</th>
<th>15 nm</th>
<th>10 nm</th>
<th>5 nm</th>
<th>3 nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solution A</td>
<td>H₂O</td>
<td>79 ml</td>
<td>79 ml</td>
<td>79 ml</td>
<td>79 ml</td>
</tr>
<tr>
<td></td>
<td>HAuCl₄</td>
<td>1 ml</td>
<td>1 ml</td>
<td>1 ml</td>
<td>1 ml</td>
</tr>
<tr>
<td></td>
<td>Na₃ Citrate</td>
<td>4 ml</td>
<td>4 ml</td>
<td>4 ml</td>
<td>4 ml</td>
</tr>
<tr>
<td></td>
<td>Tannic Acid</td>
<td>10 µl</td>
<td>100 µl</td>
<td>1 ml</td>
<td>5 ml</td>
</tr>
<tr>
<td></td>
<td>K₂CO₃</td>
<td>10 µl</td>
<td>100 µl</td>
<td>1 ml</td>
<td>5 ml</td>
</tr>
<tr>
<td></td>
<td>H₂O</td>
<td>16 ml</td>
<td>15.8 ml</td>
<td>14 ml</td>
<td>6 ml</td>
</tr>
</tbody>
</table>

β Make solution A in 250 ml flask and solution B in 50 ml flask.

β Heat solution A to ~60°C using magnetic stirrer for continuous agitation. Warm solution B to 50-60°C.

β Add solution B to solution A quickly with continuous stirring. Addition must be rapid for uniform colloid sizing.

β Continue heating to simmering (steaming but not boiling) and stir rapidly until the color is completely changed as shown below.

<table>
<thead>
<tr>
<th>Target Size</th>
<th>Initial Color</th>
<th>Time to Change</th>
<th>Final Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 nm</td>
<td>Gray-green</td>
<td>1 hour</td>
<td>Purple-red</td>
</tr>
<tr>
<td>10 nm</td>
<td>Wine-red</td>
<td>1 hour</td>
<td>Orange-red</td>
</tr>
<tr>
<td>5 nm</td>
<td>Brown-red</td>
<td>Immediate</td>
<td>Brown-red</td>
</tr>
<tr>
<td>3 nm</td>
<td>Brown</td>
<td>Immediate</td>
<td>Brown</td>
</tr>
</tbody>
</table>

β The solution is then heated to a full boil and allowed to cool.
Appendix B

Fig. 30. Heat flux versus superheat for basefluid.

Fig. 31. Heat transfer coefficient versus heat flux for basefluid.
Fig. 32. Heat flux versus superheat for 15 nm gold nanofluid (1st set).

Fig. 33. Heat transfer coefficient versus heat flux for 15 nm gold nanofluid (1st set).
Fig. 34. Heat flux versus superheat for 15 nm gold nanofluid (2nd set).

Fig. 35. Heat transfer coefficient versus heat flux for 15 nm gold (2nd set).
Fig. 36. Heat flux versus superheat for 15 nm gold nanofluid (3rd set).

Fig. 37. Heat transfer coefficient versus heat flux for 15 nm gold nanofluid (3rd set).
Fig. 38. Heat flux versus superheat for 3 nm gold nanofluid on machined surface.

Fig. 39. Heat transfer coefficient versus heat flux for 3nm gold nanofluid on machined surface.